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for the period
November 1981 to
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Low-Cost Insulator

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Author:
E. B. Toscano

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Air Force
Rocket Propulsion
Laboratory

Air Force Space Technology Center
Space Division, Air Force Systems Command
Edwards Air Force Base,
California 93523

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FOREWORD

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ERNEST B. TOSCANO
Project Manager

THOMAS J. C. CHEW
Chief, Propellant Laboratory Section

FRANCISCO Q. ROBERTO
Chief, Propellant Development Branch

FOR THE DIRECTOR

EUGENE G. HABERMAN
Director, Solid Rocket Division

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

Ablative materials serve an important function in aerospace technology. They protect aerodynamic surfaces, propulsion structures, and ground equipment from the very high temperatures and the velocity of the gases in the exhaust. This paper describes the current Air Force Rocket Propulsion Laboratory's (AFRPL) effort to evaluate an ablative coating based on a low-cost polymer and a low-cost filler. The objective of this project was to develop a low-cost, ablative/insulating material for routine application to protect costly test facilities. Experiments were conducted at the AFRPL using the standard 15-pound Ballistic

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Test and Evaluation System (BATES) solid propellant rocket motors containing aluminized propellants. This type of propellant produces an erosive exhaust gas, which is the best condition for evaluating the ablative coating formulations. Other motors were also used for evaluation purposes, such as Peacekeeper Stages I, II, and III; Short-Length Super High Internal Pressure-Producing Orifice (HIPPO); Super BATES; and the STS-5 Space Shuttle launch. The ablative samples were tested, evaluated, and compared to commercially available ablative materials under the same conditions. It was found that the low-cost ablative/insulating materials withstood the high temperature exhaust as well as, or better than, the commercially available ablative products. Using the best candidate, the Low-Cost Insulator in the one-gallon mix, cost approximately $13.00 compared to $147.00 for the same amount of the commercially available ablative material, which is a $134.00 per-gallon savings. The AFRPL ablative material can be processed in the field for easy application, and the material cures at ambient temperature.

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LOW-COST INSULATOR

I. INTRODUCTION.

1.1. Background.

When conducting experiments with rocket motors, an insulating material is applied to structures that are in the vicinity of the rocket motor's exhaust to protect them from the extreme conditions rocket motors produce so the structures can be re-used. This insulating material is costly. It is fast becoming a limiting factor in the type and amount of rocket motor firings a facility can consider, when experimenting with propellants and rocket motors, or when launching a rocket through the air or into space.

Several requirements existed for implementing this project to develop a low-cost insulator. The cost incurred by the government was of major concern, as the cost of some of the insulating materials ranged from $100 to $200 per gallon. The requisition period was too long--approximately six months. The shelf-life of the material was too short. Another major concern was the hazards to personnel that are involved when using materials containing asbestos, a known carcinogen. To alleviate these concerns, and to comply with Air Force Occupational Safety and Health Standard 161-4, the Air Force Rocket Propulsion Laboratory (AFRPL) began searching for viable alternatives by initiating an in-house project to develop an insulating material.

Could inexpensive, easy to mix, and readily available ingredients be found to replace the present insulating materials? After extensive formulation, analyses, and experimentation, it was revealed that an effective insulation was possible using an inexpensive filler mixed with a polymer binder, curing agent, and cure catalyst. And, the low-cost insulator, Toscanite, is presently in use. This report documents the processes conducted to develop this material.

1.2. Objectives.

The objective of this project was to develop an easily processable and low-cost insulating material that could be mixed in the field. The material must be durable enough to withstand the ultra high temperatures of the exhaust produced by any rocket motor in any environment. As the material must be processable in the field for easy application, it must cure at ambient temperature. The cost of the material must also compete with insulation materials already available to the government. The cost per gallon should be \$25, maximum. This meant that ingredients should be of general purpose in order to be purchased in bulk, thus reducing the cost. The ingredients must not contain asbestos nor be hazardous in any way.

1.3. Scope.

A three-phase experimental program was conducted to fulfill the project's objectives. In Phase I, several 50-gram mixes were made to determine cure time and physical properties of the material. Under Phase II, mixes selected from the 50-gram mixes were scaled up to the 500-gram size. The 500-gram mixes were placed in the direct line of the exhaust of 15-pound and 70-pound ballistic test and evaluation system (BATES) solid propellant rocket motors. The objectives of Phase III were to scale up the more successful formulations from Phase II to the one-gallon size and to evaluate these selected formulations to make the final choice.

2. EXPERIMENTAL PROCEDURES.

2.1. Basic Approach.

In Phase I, a series of 50-gram mixes was made to establish a cure time. ARCO Chemical Company's hydroxy-terminated polybutadiene (HTPB-R45HT) was the polymer binder used in all of the mixes. Isophorone diisocyanate (IPD), the curing agent containing the isocyanate groups, was used to react with (cure) the hydroxyl group in the polymer. One drop (10 micro liters) per 50-grams of dibutyltin dilaurate (DBTDL) was used as the cure catalyst. In order to achieve the desired maximum cure time of two hours, the isocyanate-to-hydroxyl ratio (NCO/OH) was varied from 0.8 to 1.5.

2.1.1. Fillers.

Several fillers were selected to try to minimize the cost. The fillers selected were carbon black, sand, glass beads, and graphite. In all of the mixes, the level of filler was varied to determine what amount would provide the best possible protection.

2.1.2. Additives.

In Phase II, an antioxidant and a flame suppressant were added to the 500-gram mixes. As the insulating material would most likely be applied to surfaces that would be exposed to the elements, the insulation must be protected from ozone oxidation for better weathering capabilities and for added protection from extreme heat. The antioxidant selected was Sherwin Williams Chemical Corporation's methylene bis(tertiary butyl phenol) (CAO-14), and the flame suppressant was lithium fluoride (LiF). The antioxidant and flame suppressant had no effect on the cure of the insulation. These 500-gram mixes were then subjected to the exhaust of 15-pound and 70-pound BATES solid propellant rocket motors.

2.1.3. Scale-up Experiments.

From the results of the 500-gram mixes, several successful candidates were selected and scaled up to the one-gallon mix in Phase III. The candidates selected were carbon black, sand, and glass beads. In this phase, the level of fillers was also varied to determine the best possible candidate for the final product. The one-gallon mixes were subjected to the exhaust of solid and liquid propellant rocket motors. The rocket motors used were the Peacekeeper Stages I, II, and III; super BATES; Short-Length Super High-Internal Pressure-Producing Orifice (HIPPO); and the Space Shuttle, Columbia. The primary goal of subjecting the insulation to extreme conditions was to produce an insulation that would endure the intense heat (theoretically 4000°F to 6000°F) and hot gases produced by the exhaust of full-scale motors and still protect the surface to which it was applied.

2.1.4. Mixing Procedure.

To simulate field conditions, the mixing procedure was kept to a simple format. The mixing was done completely by hand, using simple tools. A container large enough to hold the desired amount of insulation material was used as the mixing bowl. The mixer was a half-inch drill motor. A mixing blade was made from a piece of 3/8-inch stainless steel tubing, with a 90° bend at the end.

The mixing procedures were as follows:

1. Pre-weigh the following:

    a. Polymer (binder), HTPB R45HT.

    b. Curing agent, IPDI.

    c. Solids (fillers).

    d. Cure catalyst, DBTDL.

    e. Antioxidant, CAO-14. *

    f. Flame suppressant. *

    * These ingredients can be omitted.

2. Thoroughly mix the polymer and the curing agent.

3. Add all of the solids (filler), and mix thoroughly.

4. Add the cure catalyst, and mix thoroughly.

If a flame suppressant and an antioxidant are used, follow steps 5 and 6.

5. Add the antioxidant, and mix thoroughly.

6. Add the flame suppressant, and mix thoroughly.

The weight percentages of the ingredients are calculated as follows:

1. The NCO/OH ratio is predetermined at 1.5.

2. The percentage of flame suppressant is predetermined at 2% of the mix.

3. The percentage of antioxidant is predetermined at 1% of the mix.

4. The percentage of solids (fillers) can also be predetermined. The percentage will vary depending on which filler is used. For example, the percentage is 87% sand with 2% flame suppressant and 1% antioxidant. This equates to 90%.

5. The percentage of cure catalyst is predetermined at 0.05% of the mix or 10 µl-per-50 grams.

The following method is used to determine the amount of ingredients used in the formulation of this mix, which is a 500-gram mix with 90% filler:

To calculate the weight of the polymer, a fraction is formed. The equivalent weight of the polymer is taken as the numerator. The equivalent weight of the curing agent is multiplied by the NCO/OH ratio. The resulting product is then added to the equivalent weight of the polymer. This sum becomes the denominator. The resulting fraction is then multiplied by the percentage of binder. In this example, it is 10%. This number is multiplied by the total weight of the mix. In this example, it is 500 grams.

$$\frac{1350}{(112)(1.5) + 1350} = 0.889$$

$$(0.889)(10\%) = 0.0889 \qquad \text{(EQ. 2.1.4-1)}$$

$$(0.0889)(500 \text{ grams}) = 44.5 \text{ grams polymer}$$

To calculate the weight of the curing agent, a fraction is formed. The equivalent weight of the curing agent is multiplied by the NCO/OH, forming the numerator. The denominator is the equivalent weight of the curing agent multiplied by the NCO/OH. The resulting product is added to the equivalent weight of the binder. The resulting fraction is then multiplied by the percentage of binder, and that result is multiplied by the total weight of the sample.

$$\frac{(112)(1.5)}{(112)(1.5) + 1350} = 0.110$$

$$(0.110)(10\%) = 0.011 \qquad \text{(EQ. 2.1.4-2)}$$

$$(0.011)(500 \text{ grams}) = 5.50 \text{ grams curing agent}$$

The following is an example of the ingredients, percentages, and weights of each ingredient used:

Ingredient		% Used	Weight(grams)
Polymer	R45HT	8.84	44.2
Curing Agent	IPDI	1.1	5.5
Solids	Sand	87.0	435.0
Flame Suppressant	LiF	2.0	10.0
Antioxidant	CAO-14	1.0	5.0
Cure Catalyst	DBTDL	0.05	0.3 or 50µl

2.2. Hardware.

The 500-gram mixes were cast on a steel plate 1/4 inch thick, 5 inches long, and 4 inches wide. Four thermocouples were mounted as in Figures 1 and 2, when two samples were tested simultaneously. To minimize the amount of radiant heat that would affect the thermocouples and to ensure a direct blast on the samples, a graphite adapter was used. Figures 3 and 4 illustrate the graphite adapter. The samples were mounted on a pedestal and placed 10 feet downstream of the motor. This distance was sufficient to enable the samples to experience enough flame impingement for a valid test, as illustrated by Figures 5 and 6.

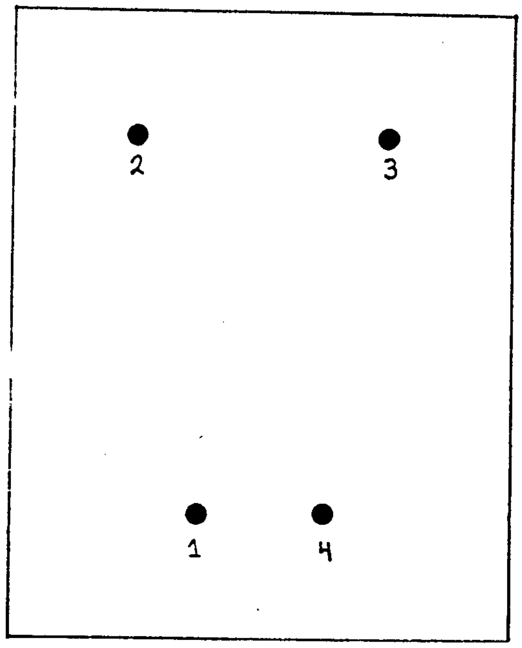

Figure 1. Thermocouple Location (Single Sample).

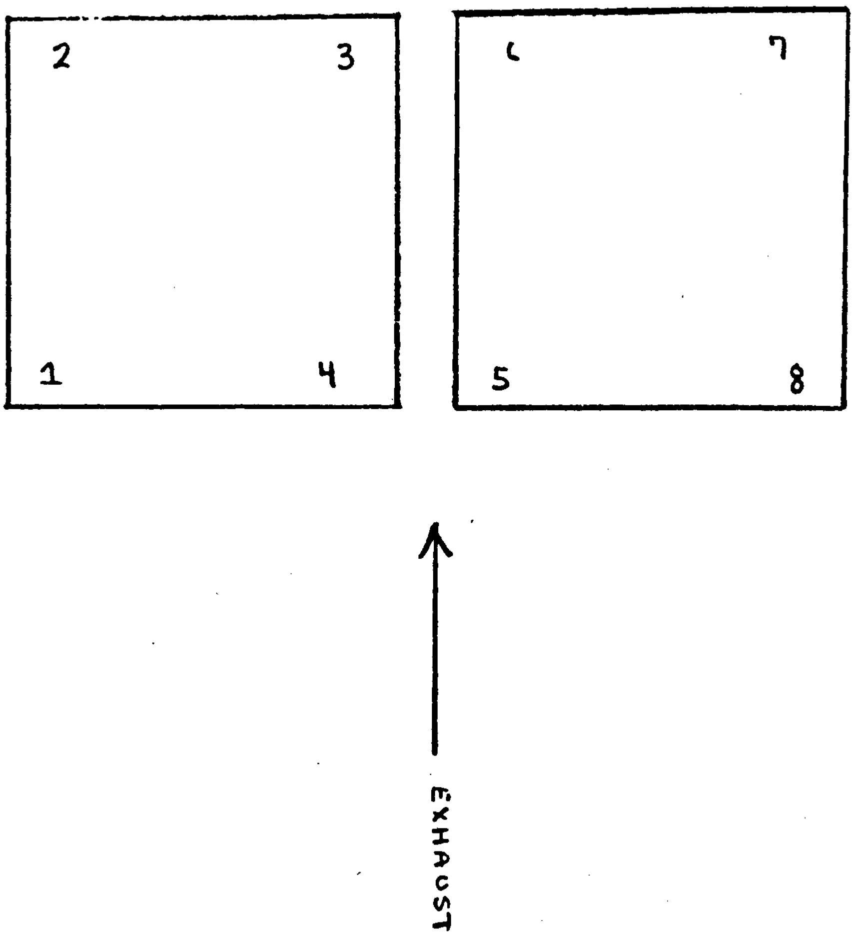

Figure 2. Thermocouple Location (Double Samples).

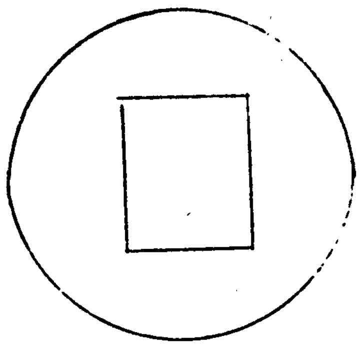

Figure 3. Graphite Billet (Single Sample).

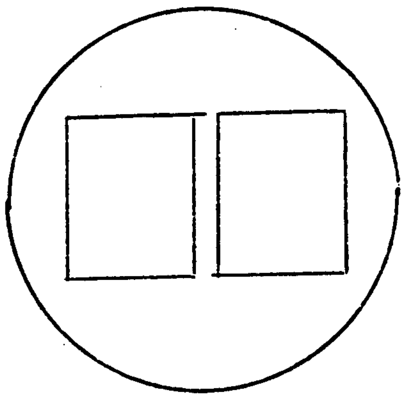

Figure 4. Graphite Billet (Double Samples).

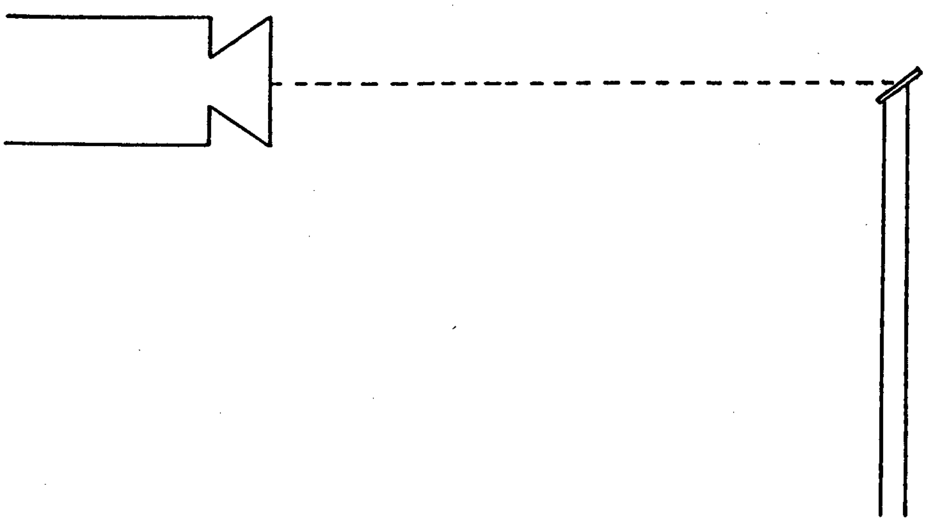

Figure 5. Experimental Configuration.

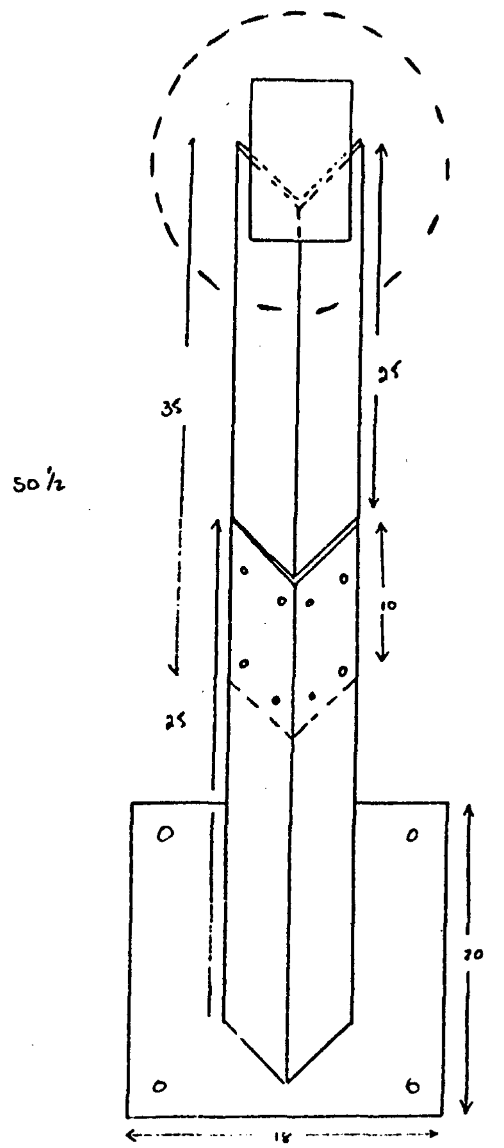

Figure 6. Mounting Pedestal.

2.3. Compatibility Experiments.

To provide adequate protection in a liquid rocket propellant environment, the material itself must not produce flame or heat. The following compatibility experiments were conducted on the samples considered for scale-up to the 500-gram mix.

1. All samples were weighed and hardness-tested (using the Shore A Hardness test) prior to the experiment. Each sample weighed approximately 4 grams. The hardness tests used were ASTM-D-1484-59 and 170-6-61, Type D.

2. RP-1, N_2H_4, UDMH, and MMH experiments were run for four hours with the sample half immersed in the liquid. The samples were removed from the liquid and allowed to dry overnight. Final weight and hardness tests were completed the following day.

3. Samples were placed in dewar, covered with liquid oxygen (LO_2), and left overnight to permit LO_2 evaporation. They were then weighed and tested for hardness.

4. Samples for N_2O_4 experiments were placed in glass vials and liquid N_2O_4 was added. Additional N_2O_4 was added as required for the four-hour test period. These samples could not be recovered.

Table I gives weight and visual observations. Figures 7 through 12 are the density-versus-solids-loading plots.

Table I. Results of Compatibility Studies.

Mix Number		Grams Wt. Change *	Visual Observation
RP-I	80-10	+0.100	No visible change (NVC)
RP-I	90-2	+0.069	NVC
RP-I	90-3-I	+0.076	NVC
RP-I	9-5	+0.095	NVC
N_2H_4	80-I	-0.024	Slight reaction when N_2H_4 added, subsided after a few minutes.
N_2H_4	80-3-I	-0.004	NVC
N_2h_4	80-10	+0.002	NVC
N_2H_4	90-5	+0.002	NVC
UDMH	90-I-I	-0.048	Soaked up liquid and sample fell apart upon addition of UDMH.
UDMH	90-3	-0.078	Some sample separation.
UDMH	90-5	+0.022	NVC
UDMH	80-10	+0.019	NVC
MMH	80-10	+0.005	NVC
MMH	90-2-I	---	Sample fell apart.
MMH	90-4	-0.20	Some sample fell off.
MMH	90-5	+0.007	NVC
LO_2	80-2-I	0	NVC in this sample overnight.
LO_2	80-3	-0.008	NVC in this sample overnight.
$L0_2$	80-10	+0.001	NVC in this sample overnight.
$L0_2$	90-5	0	NVC in this sample overnight.
N_20_4	90-5	--	Slight bubbling.**
N_20_4	80-10	--	Slight bubbling.**
N_20_4	80-4	--	Slight bubbling, fell apart.**
N_20_4	80-I-I	--	Rapid bubbling.**

*Each sample weighed approximately 4 grams.

**All of these samples swelled after two hours, and became tar after four hours.

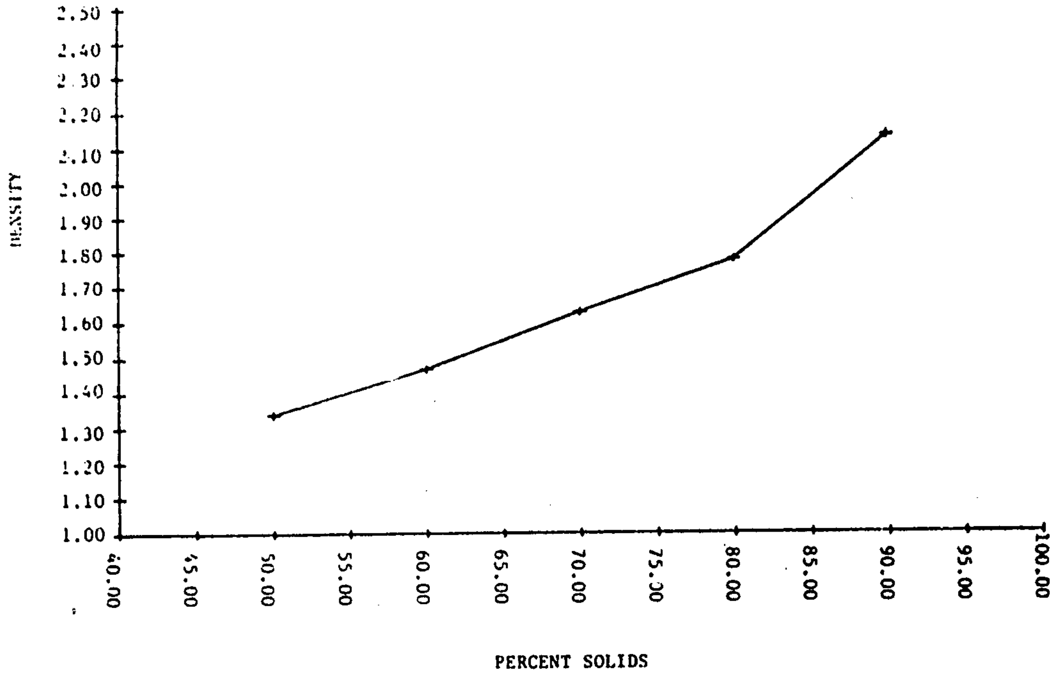

Figure 7. Density Plot, Sand, NCO/OH Ratio 1.0

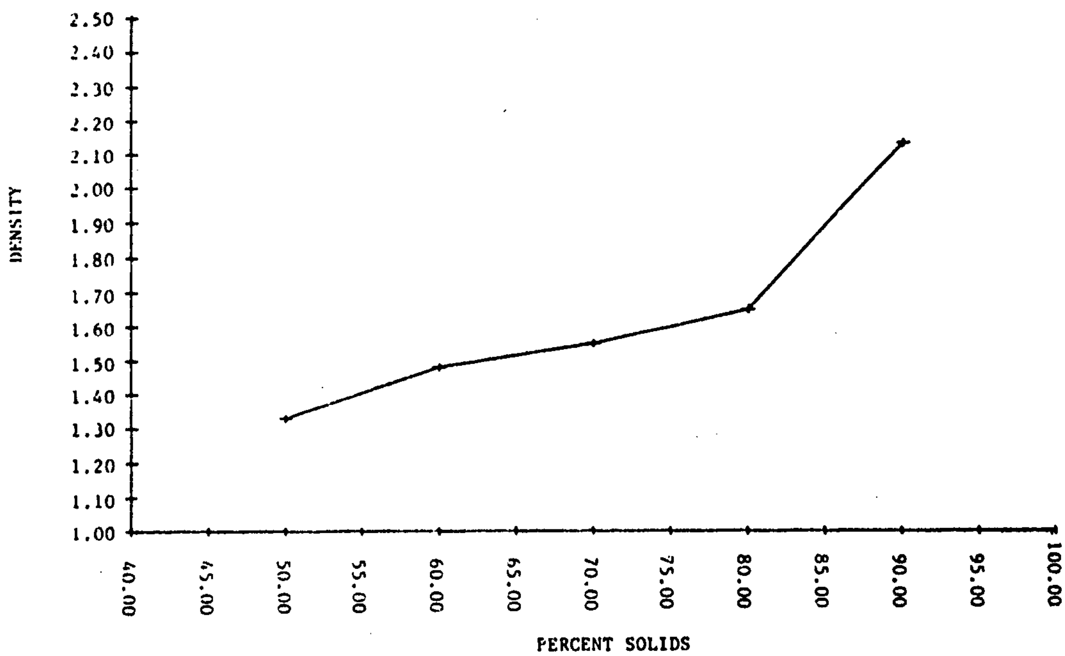

Figure 8. Density Plot, Glass Beads, NCO/OH Ratio 1.0.

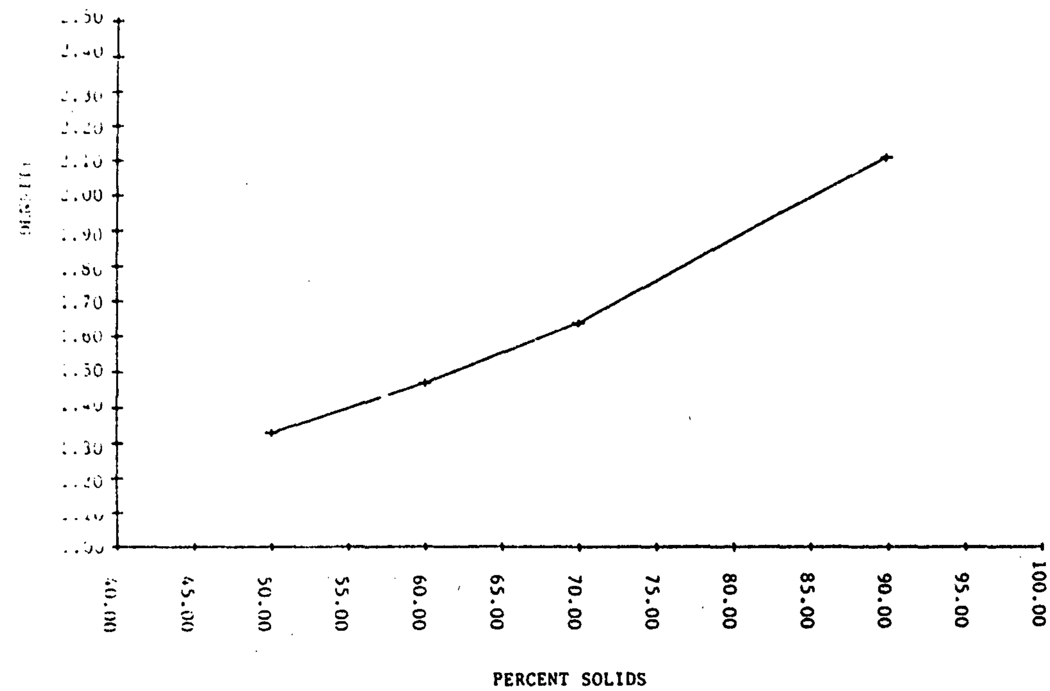

Figure 9. Density Plot, Sand, NCO/OH Ratio 0.9.

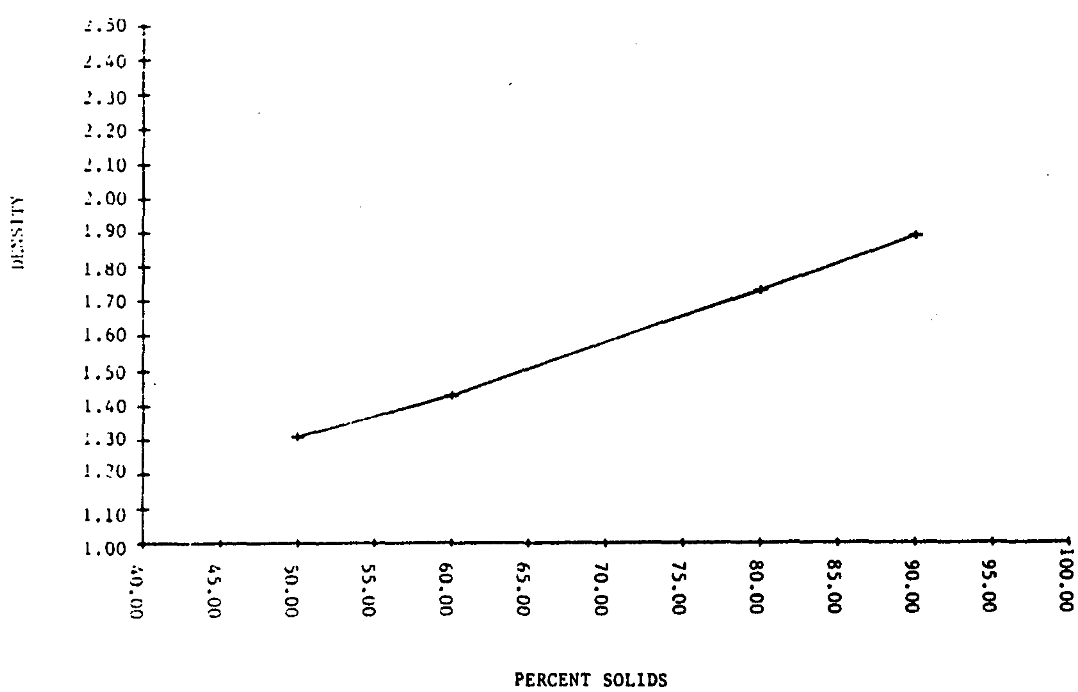

Figure 10. Density Plot, Glass Beads, NCO/OH Ratio 0.9.

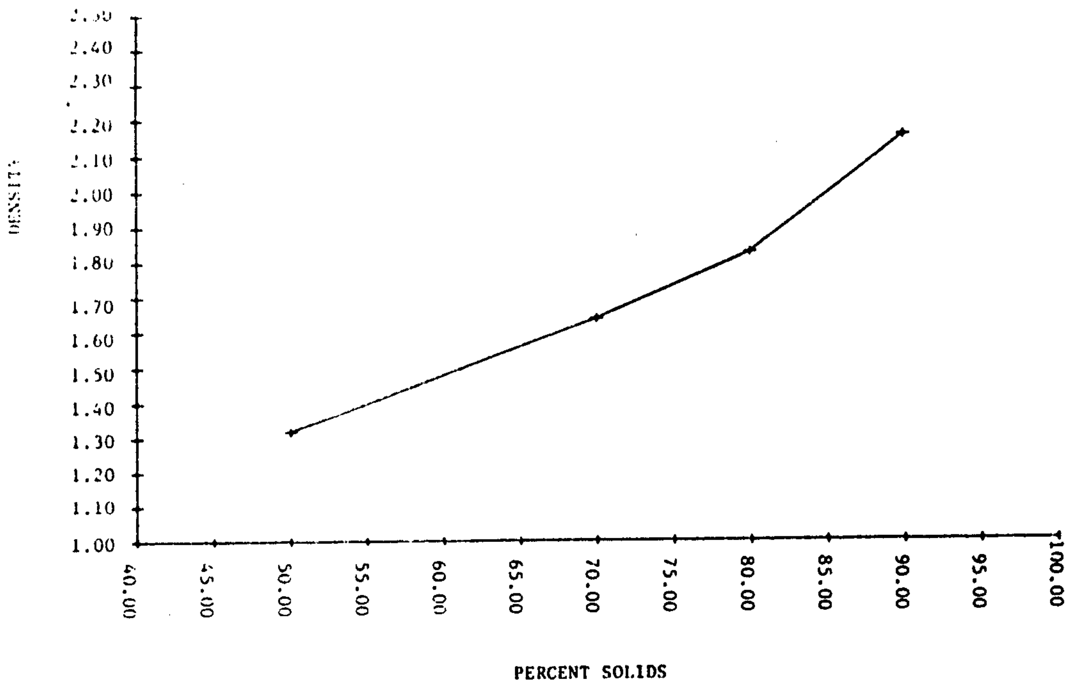

Figure 11. Density Plot, Sand, NCO/OH Ratio 0.8.

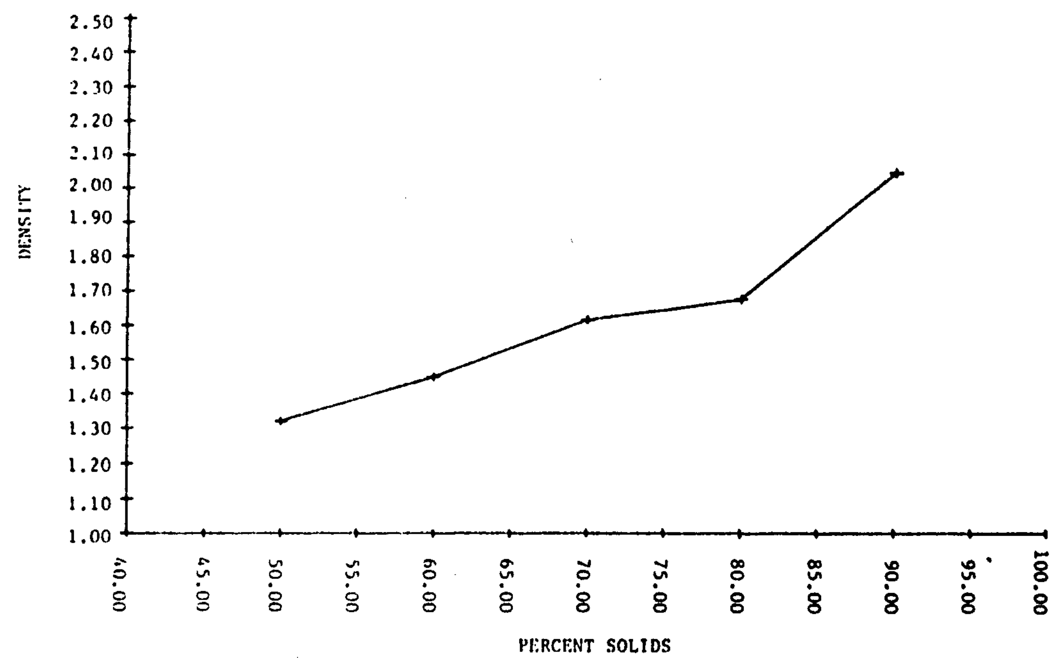

Figure 12. Density Plot, Glass Beads, NCO/OH Ratio 0.8.

Appendix A lists the results of the Shore A hardness tests and the density of the 50-gram mixes. The formulations of the 50-gram mixes can be found in Appendix B. The 50-gram formulations with additives are in Appendix C.

2.4. Experimental Projects.

Initial experiments were conducted on commercially available insulating materials. We used a Kirkhill Rubber Company product, ME Hill-Gard V-61, and AVCO's Char-Tek 59. These products were subjected to the exhaust of 15-pound BATES solid propellant rocket motors.

The 500-gram scaled up samples were subjected to 70-pound BATES solid propellant rocket motors. Most of the firings tested two samples at a time. The graphite billet was used to contain the majority of the flame on the samples. The core of the exhaust impinged on the center of both samples. Pre- and post-firing measurements were taken to determine the extent of erosion. Pre- and post-Shore A hardness tests were accomplished to determine physical changes in the material. Temperature measurements were recorded to register heat conduction through the samples.

The one-gallon scaled up mixes were subjected to the environment produced by the firing of full-scale motors. The Dow-Corning product, DC 93-058, was also subjected to the exhaust of these motors. The experiments were designed to expose the material, not only to direct exhaust impingement, but also to radiant heat. The purpose of subjecting the samples to different environments was to verify that the capabilities of our one-gallon mixes exceeded those of the commercial products.

3. EXPERIMENTAL RESULTS AND DISCUSSION.

3.1. Initial Experiments.

On this particular series of samples, Char-Tek 59 and V-61 were tested together. Other samples tested separately were those of 90% Char-Tek 59 containing 10% graphite and 50% Char-Tek 59 containing 50% graphite. On both tests that contained the Char-Tek 59 sample, the Char-Tek sample was epoxied to the steel plate and V-61 was cast around it. The other samples of Char-Tek contained graphite. The graphite was mixed into the Char-Tek material, and the cured mixture was epoxied onto the steel plate.

These samples were tested individually using 15-pound BATES solid propellant rocket motors, which produced a theoretical flame temperature of 4000°F. The samples were placed 10 feet downstream of the motor, and the exhaust impinged directly on the center of the samples. The duration of each firing lasted approximately 2.5 seconds. The samples held up very well. Figures 13 through 20 show the pre- and post-firing photographs of the samples. Figures 21 through 25 are temperature-versus-time traces.

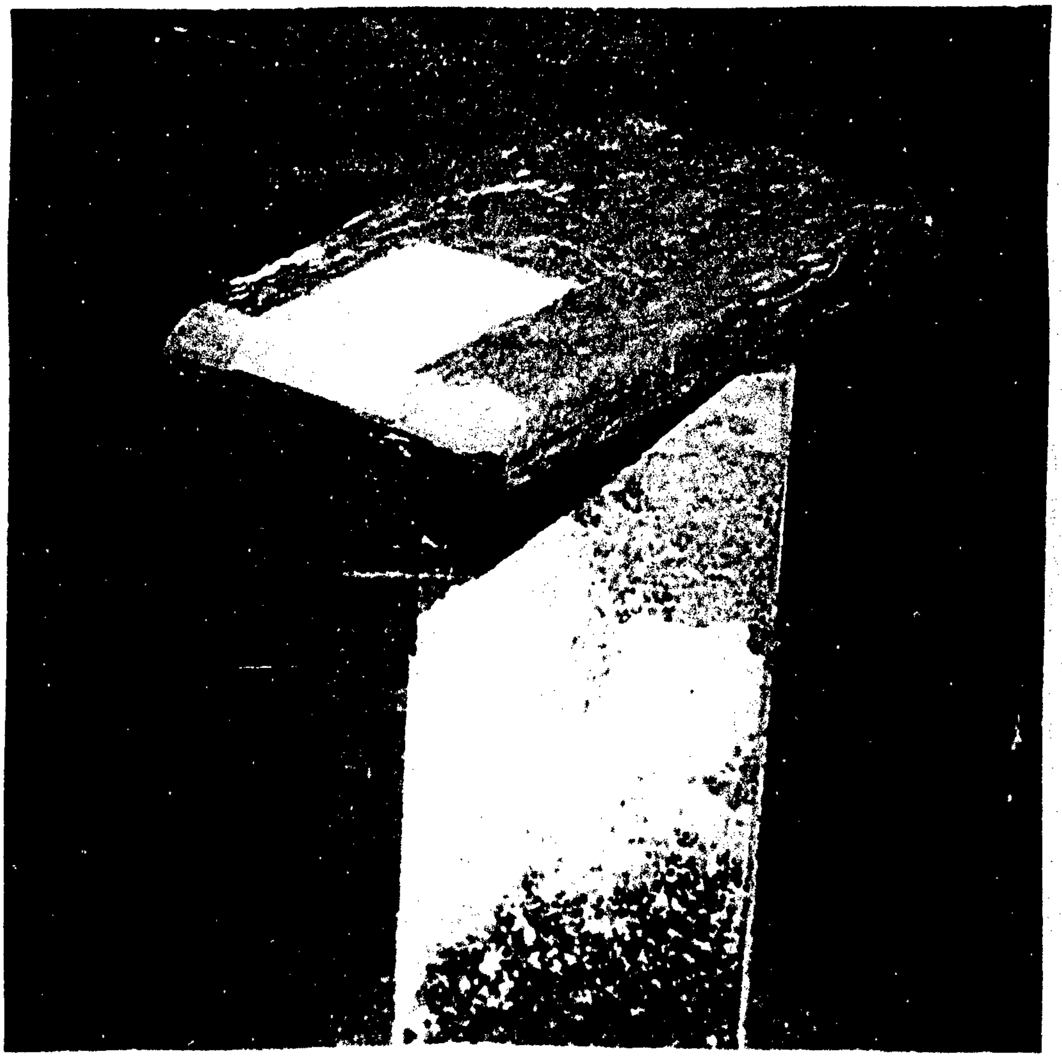

Figure 13. Char-Tek 59 and V-61 Sample, Pre Photograph.

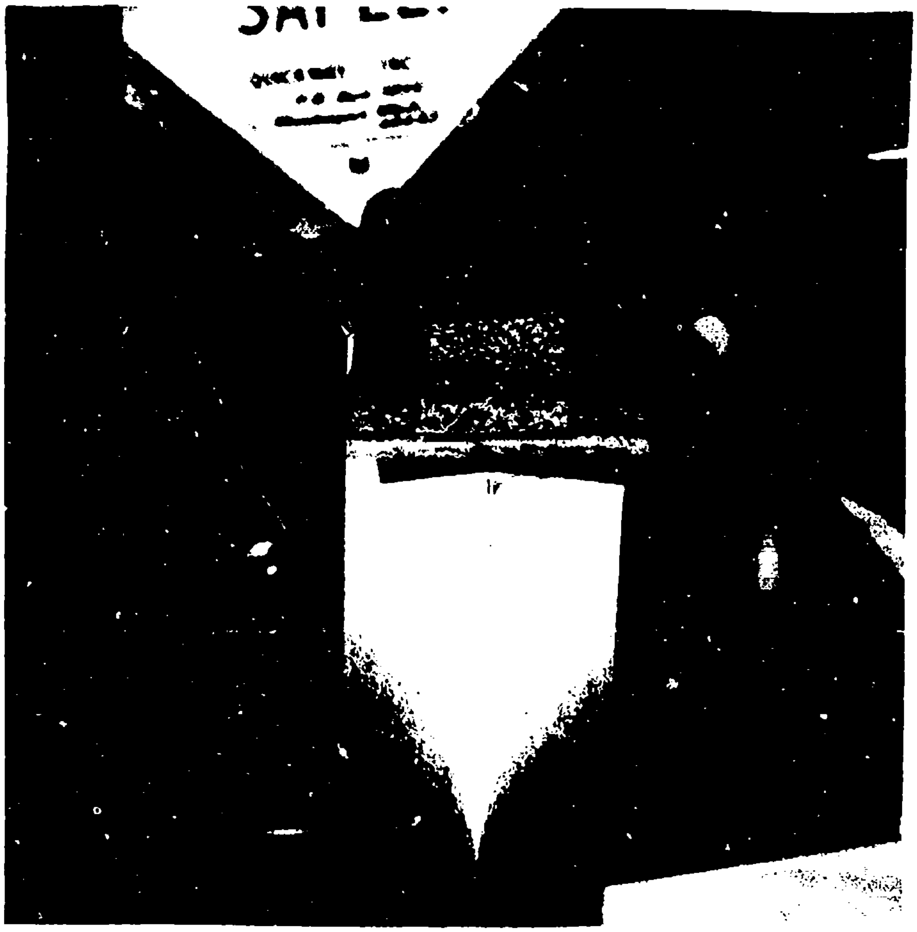

Figure 14. Char-Tek 59 and V-61 Sample, Post Photograph.

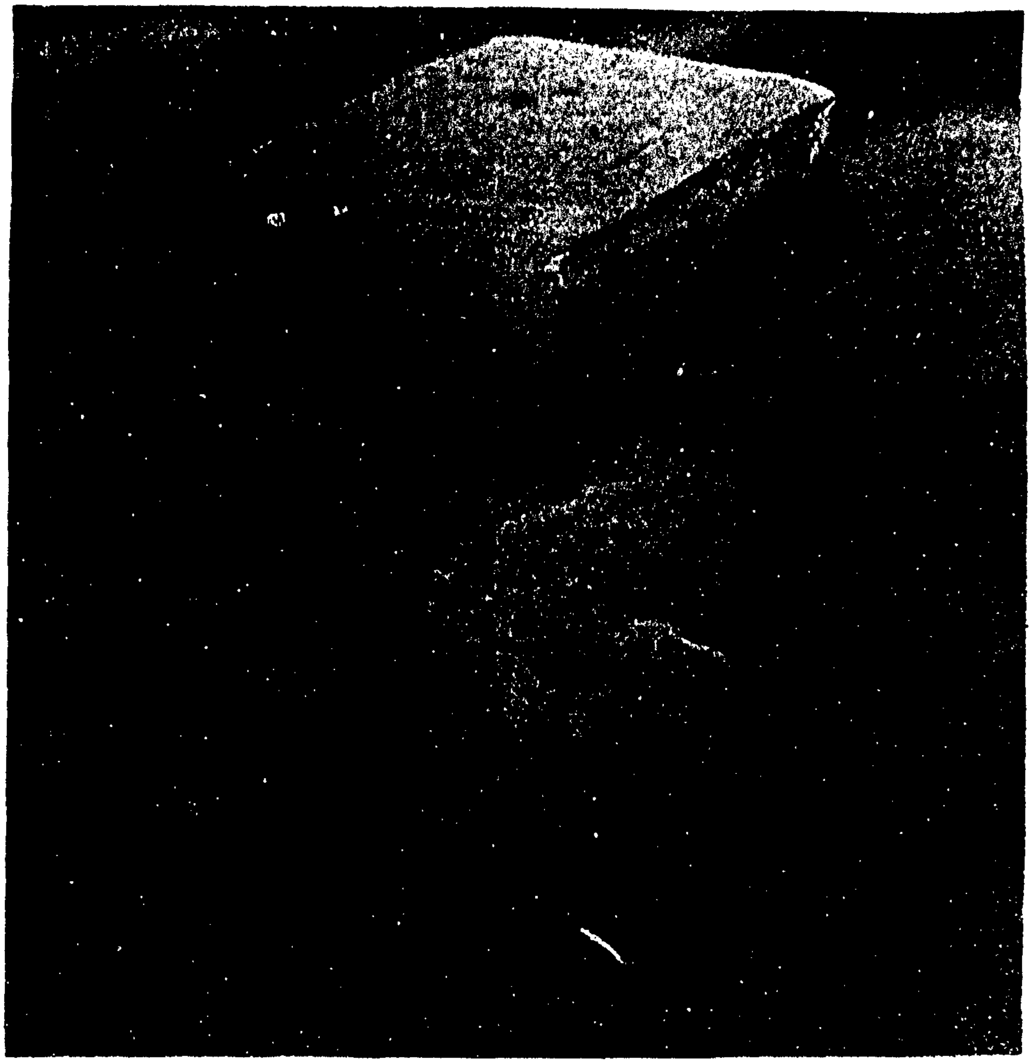

Figure 15. Char-Tek 59 Sample, Pre Photograph.

Figure 16. Char-Tek 59 Sample, Post Photograph.

Figure 17. 90% Char-Tek 59, 10% Graphite Sample, Pre Photograph.

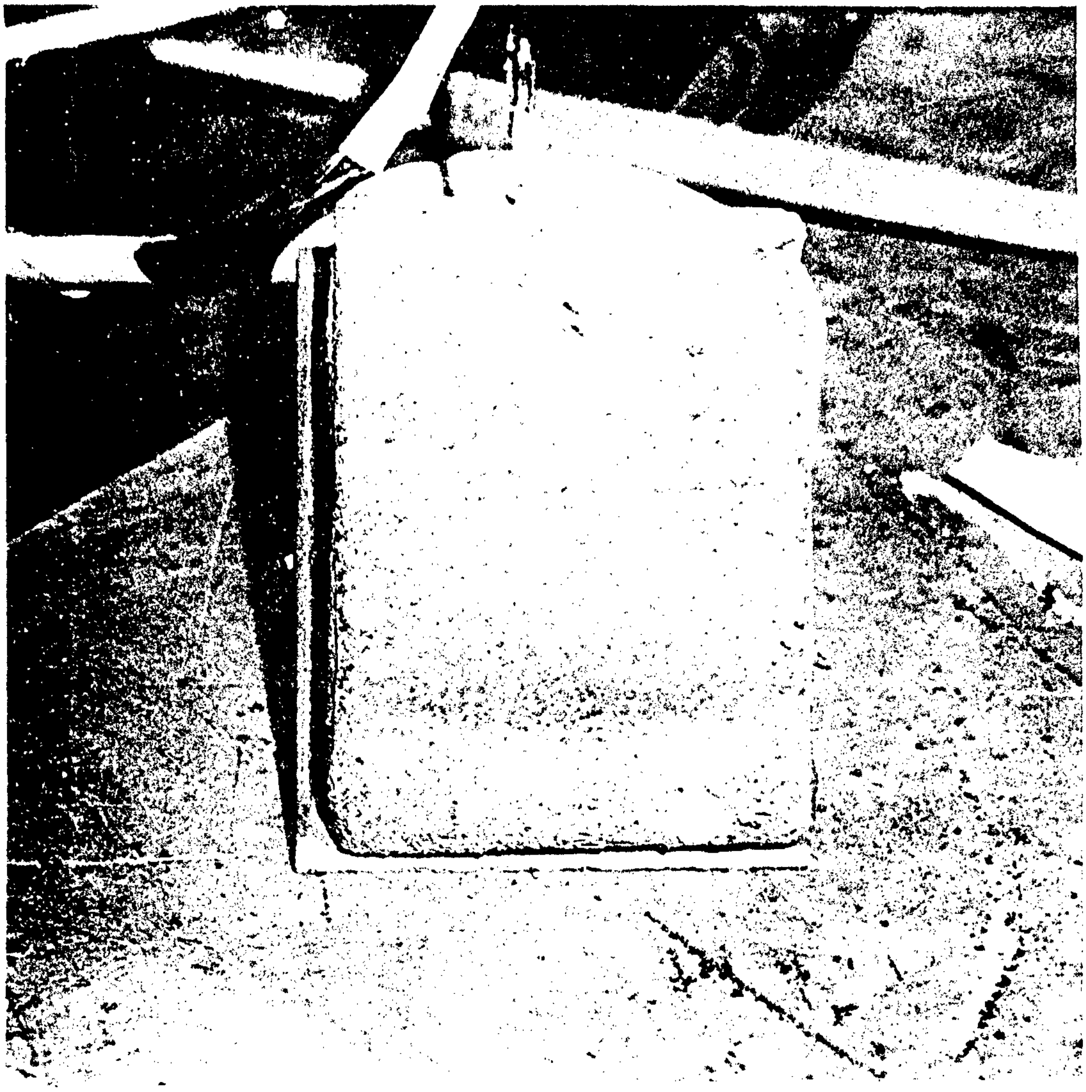

Figure 18. 90% Char-Tek 59, 10% Graphite Sample, Post Photograph.

Figure 19. 50% Char-Tek 59, 50% Graphite Sample, Pre Photograph.

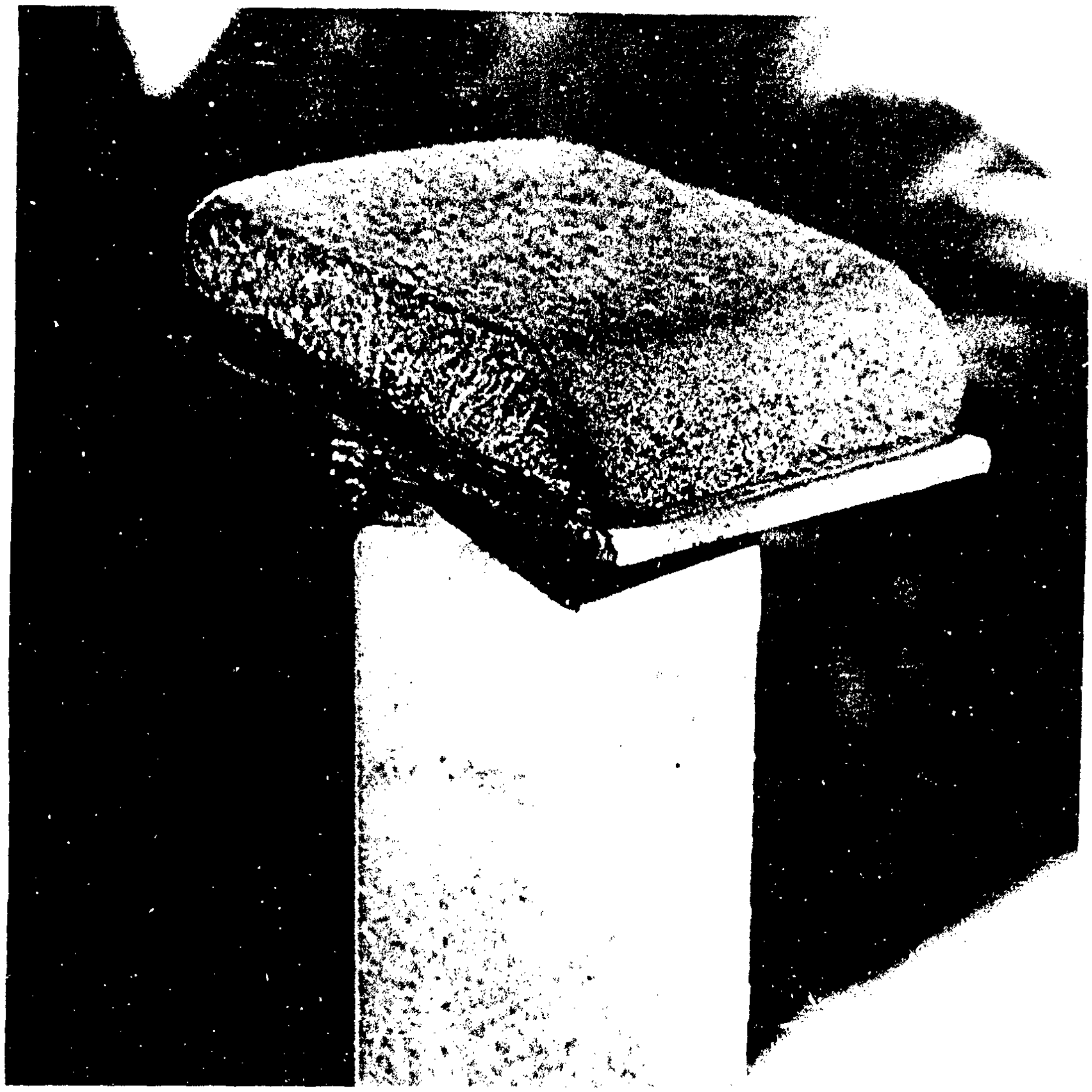

Figure 20. 50% Char-Tek 59, 50% Graphite Sample, Post Photograph.

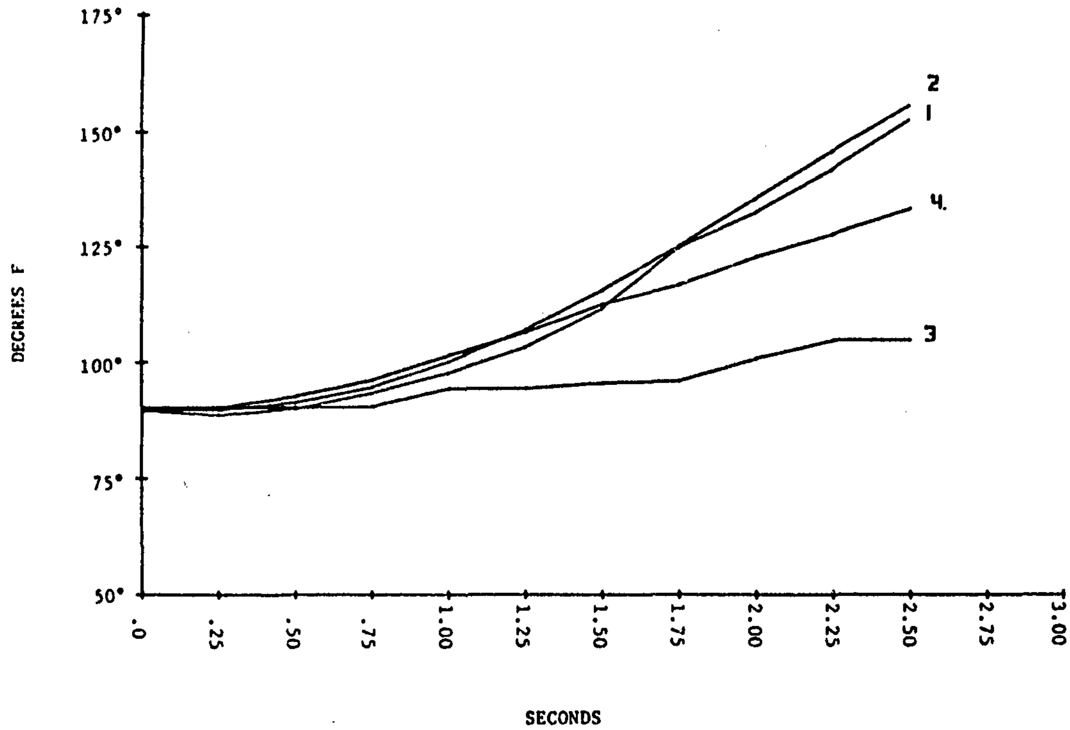

Figure 21. Char-Tek 59 and V-61 Sample, Temperature-vs-Time Trace.

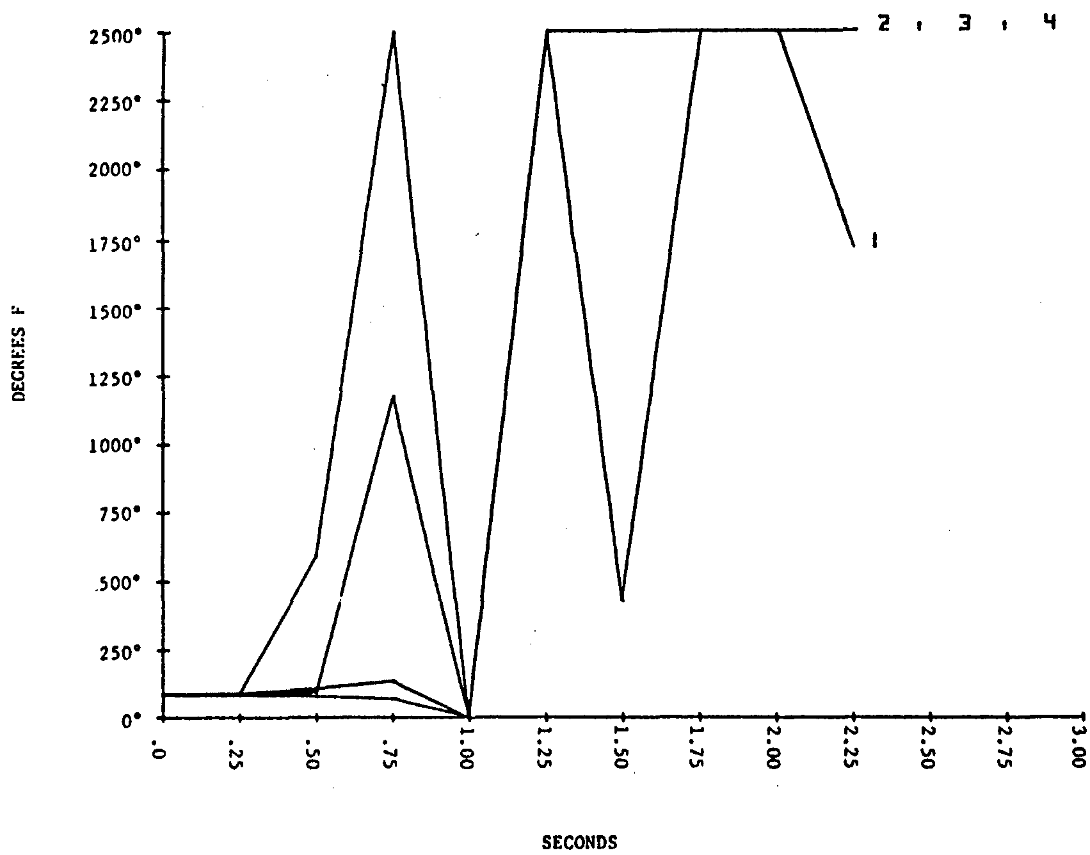

Figure 22. Char-Tek 59 and V-61 Sample, Temperature-vs-Time Trace.

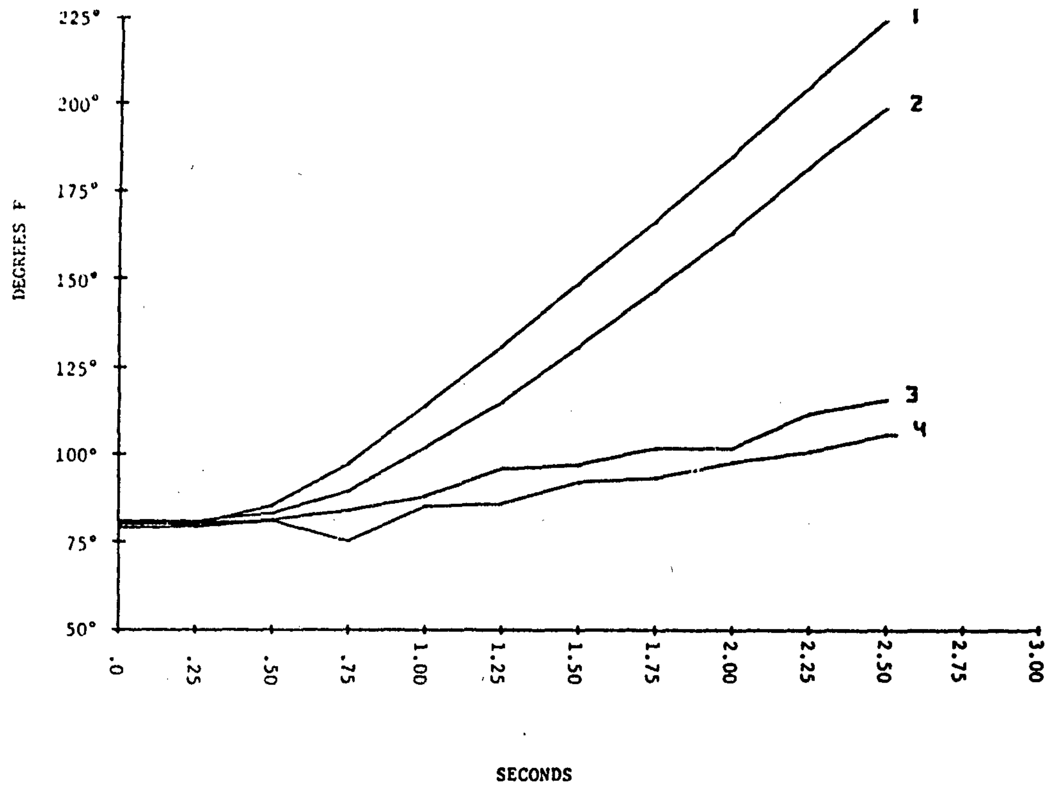

Figure 23. 90% Char-Tek 59, 10% Graphite Sample, Temperature-vs-Time Trace.

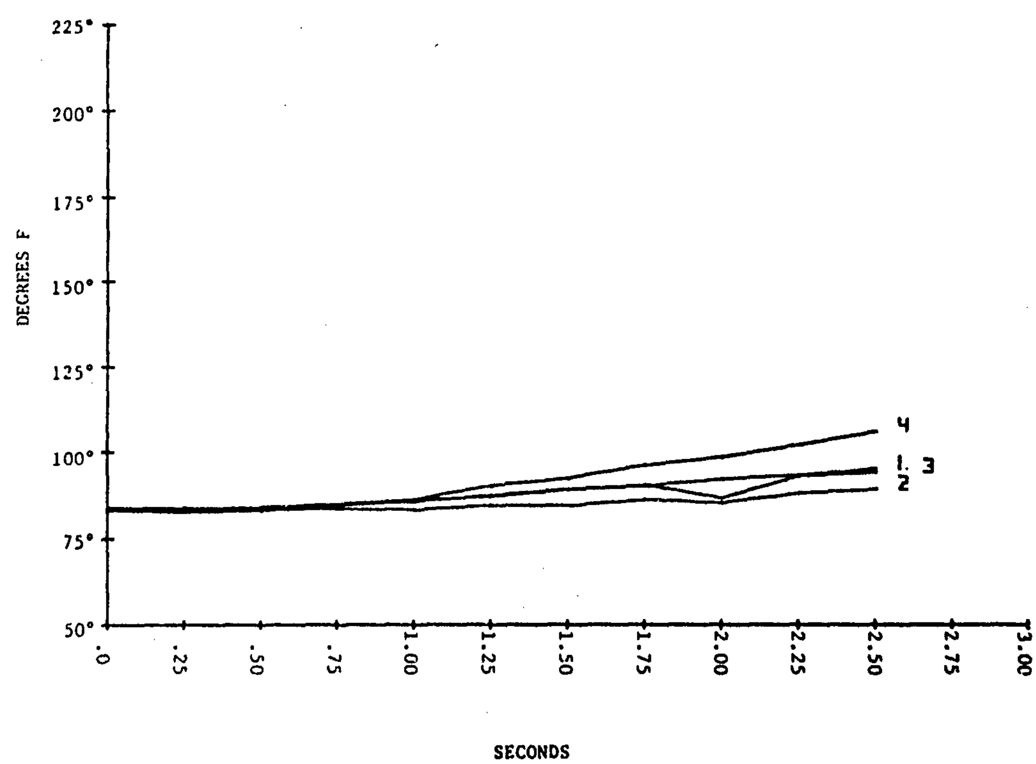

Figure 24. 50% Char-Tek 59, 50% Graphite Sample, Temperature-vs-Time Trace.

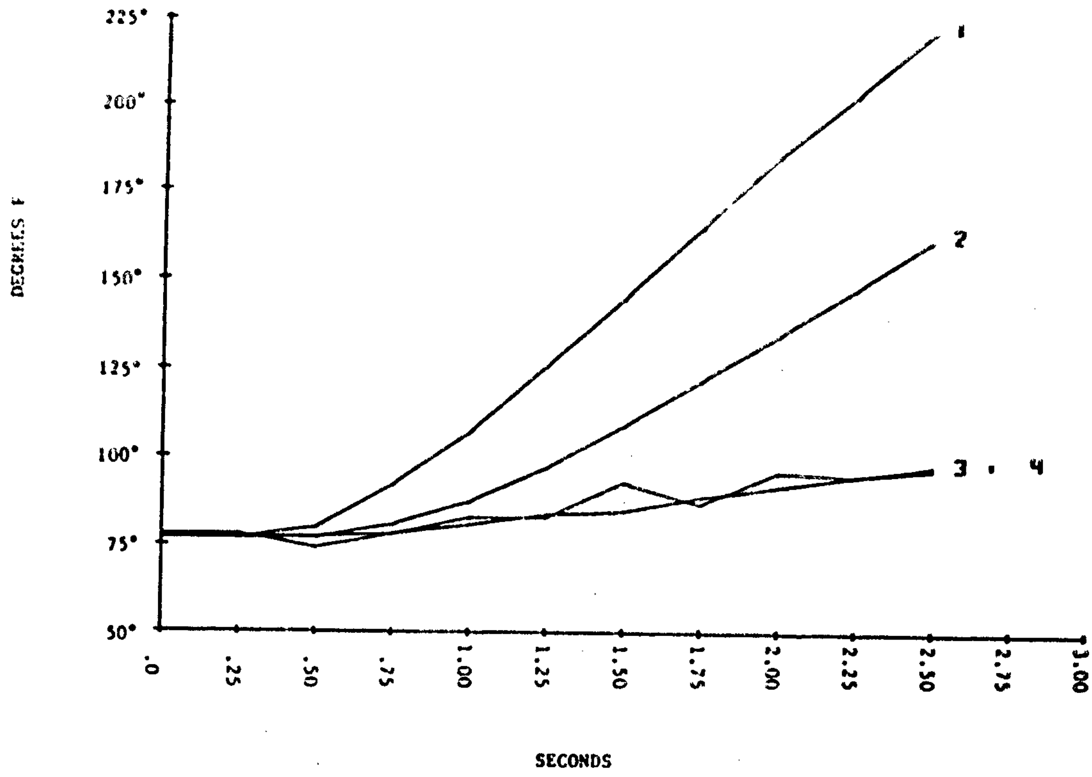

Figure 25. Char-Tek 59 Sample, Temperature-vs-Time Trace.

3.2. 500-Gram Sample Experiments.

During Phase II, in this particular series of experiments, several firings tested two samples at a time. To minimize the amount of radiant heat affecting the thermocouples and to ensure a direct blast on the samples, a graphite billet was modified to accommodate these requirements. The exhaust was produced by a 70-pound BATES solid propellant rocket motor.

The samples were instrumented with thermocouples to record heat conduction. The distance between the samples and the motor was 10 feet, which was sufficient to enable the samples to experience direct flame impingement. Refer to Figures 1 and 2 for thermocouple locations and to Figure 5 for the set-up of the experiment.

A flame suppressant (LiF) and an antioxidant (CAO-14) were added to some of the samples. The comparison of samples containing LiF and CAO-14 to those without indicated whether or not those additional ingredients increased the material's ability to more effectively withstand rocket exhaust and resist ozone oxidation.

The duration of the firings ranged from 3.75 to 9.5 seconds. Most of the samples held up very well for the first 4 seconds. No debonding of any of the material was evident. Some of the thermocouple data was erratic. This was due to erosion of the sample's leading edge, which allowed heat to be transmitted through the steel plate. At this point, it was thought the material that promised to fulfill the project's objectives was the carbon black. It was thought that the sand material also had some limited application.

Table 2 lists the samples tested and the results of the measurements taken before and after the experiment. The temperature-versus-time traces are presented in Figures 26 through 38. Pre- and post-firing photographs are presented in Figures 39 through 52.

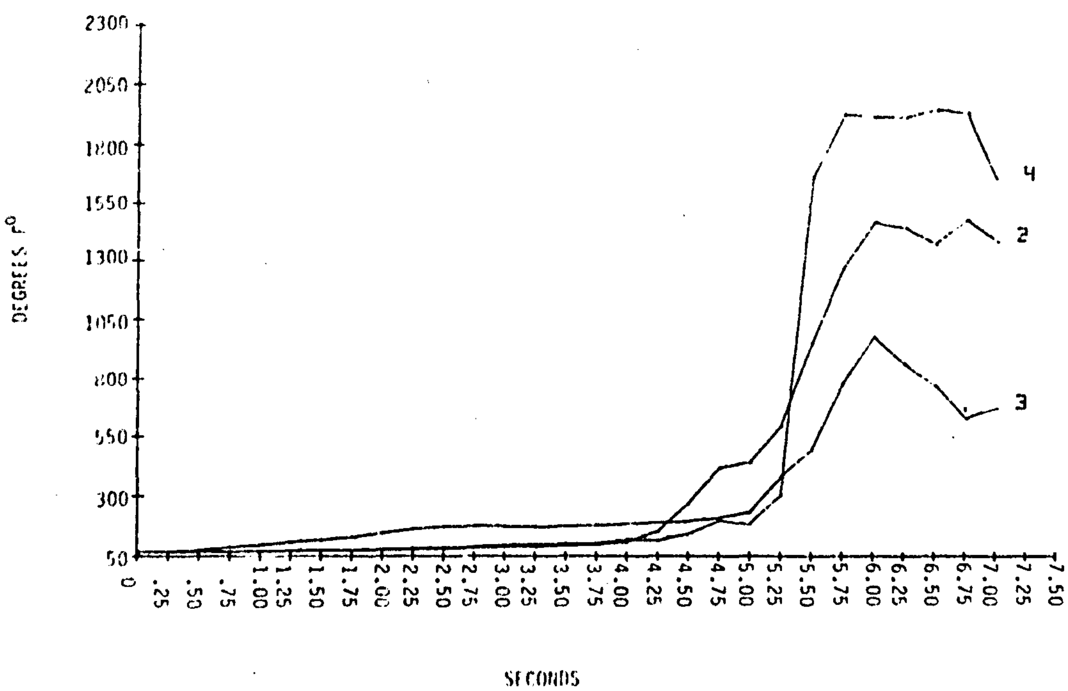

Figure 26. 80% Sand, LiF, CAO-14 Sample, Temperature-vs-Time Trace.

Table 2. Results of 500-Gram Experiments.

Mix Number	Composition	Pre-Shore A	Post-Shore A	Pre-Measurement(in)	Post-Measurement(in)	Material Loss(%)	Erosion Rate(ips)
80-1-1.5B	80% Sand (LiF)(CAO-14)	76	65	0.872	0.383	56	0.07
80-5-1.5B	80% Graphite (LiF)(CAO-14)	72	60	1.04	0.240	82	0.315
90-2-1.5A	90% Glass Beads	82	*	0.783	*	*	*
80-5-1.5A	80% Graphite	73	*	1.14	*	*	*
30-4-1.5A	30% Carbon Black	37	60	0.904	0.271	71	0.08
80-1-1.5A	80% Sand	67	*	0.846	*	*	*
20-4-1.5B	20% Carbon Black(LiF)(CAO-14)	44	50	1.06	0.766	28	0.06
25-4-1.5B	25% Carbon Black (LiF)(CAO-14)	43	51	1.266	1.02	19	0.05
90-2-1.5B	90% Sand (LiF)(CAO-14)	66	76	0.789	0.277	65	0.07
90-1-1.5A	90% Sand	75	73	0.823	0.255	71	0.08
80-1-1.5	80% Sand (LiF)(CAO-14)	70	58.2	0.821	0.267	67	0.15
25-4-1.5A	25% Carbon Black	34	54	0.877	0.100	89	0.21
35-4-1.5A	35% Carbon Black	40	*	1.034	0.663	37	0.08

*No measurements taken.

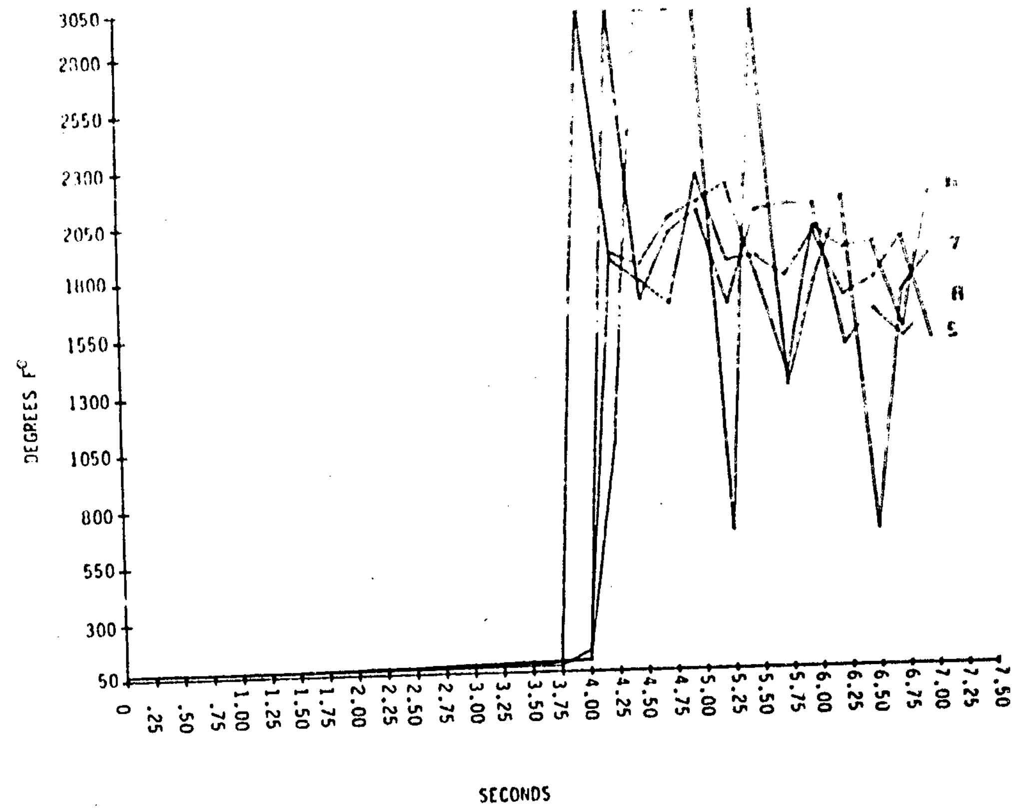

Figure 27. 80% Graphite, LiF, CAO-14 Sample, Temperature-vs-Time Trace.

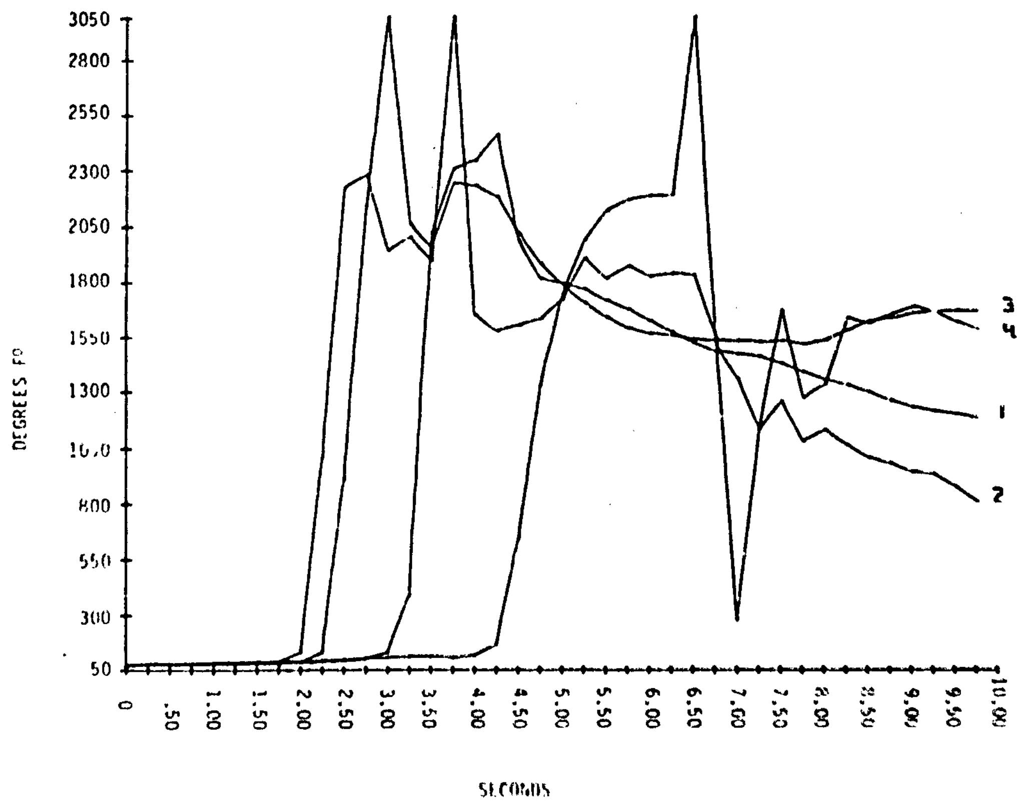

Figure 28. 90% Glass Beads Sample, Temperature-vs-Time Trace.

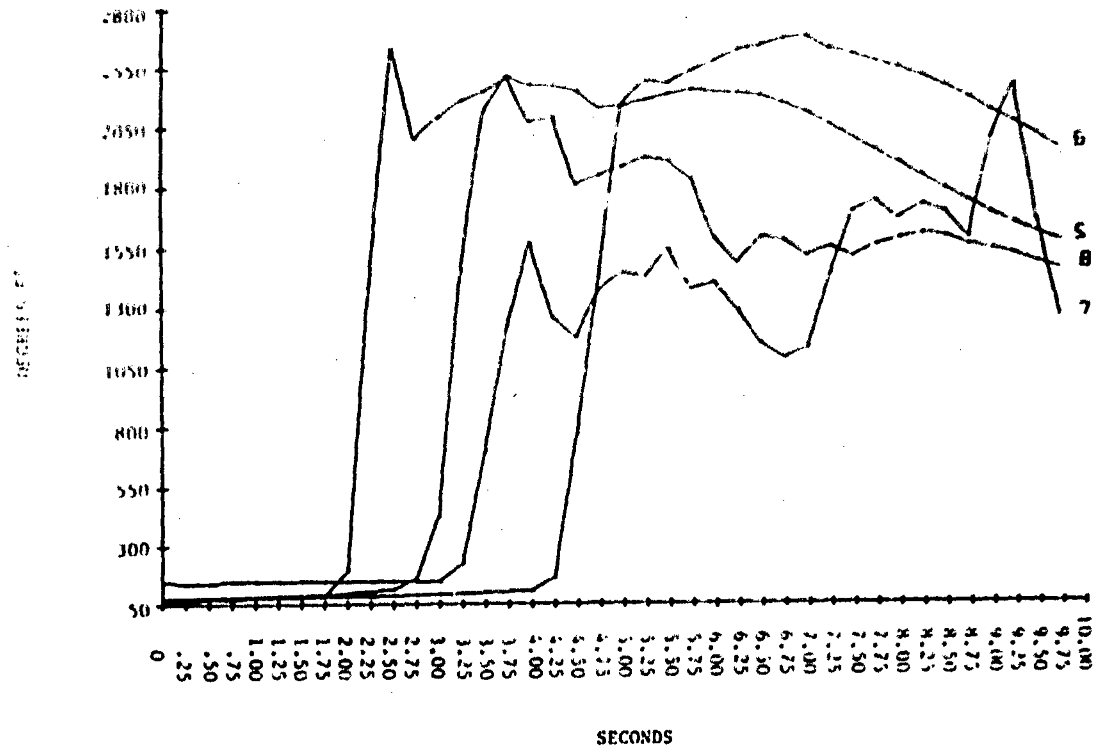

Figure 29. 80% Graphite Sample, Temperature-vs-Time Trace.

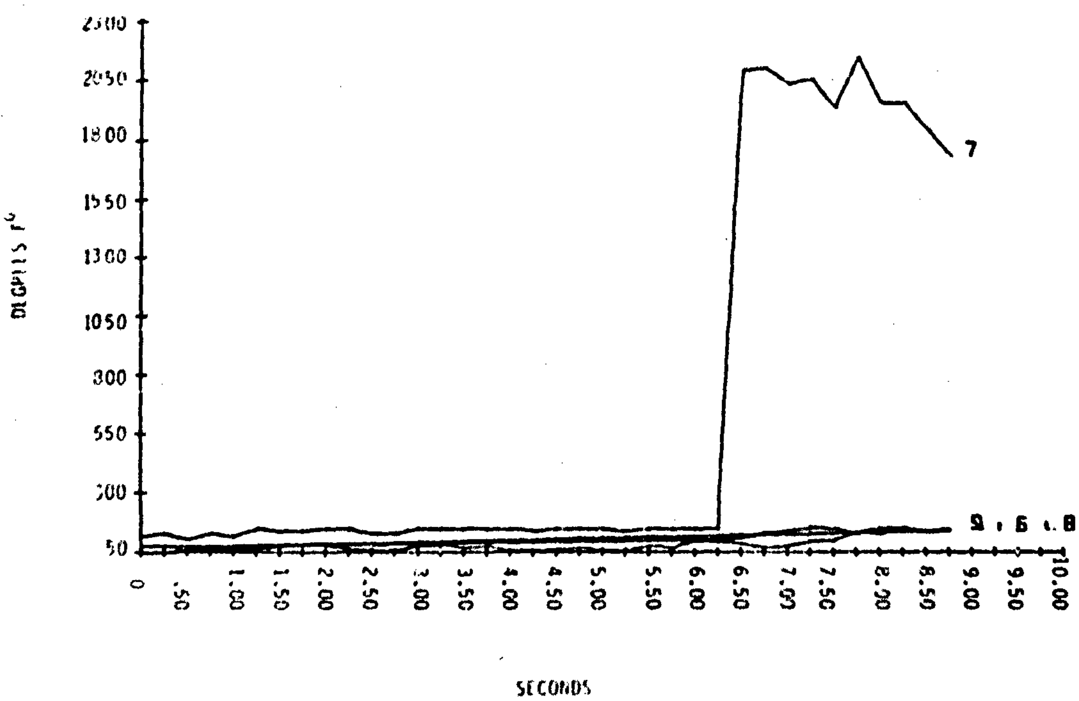

Figure 30. 80% Sand Sample, Temperature-vs-Time Trace.

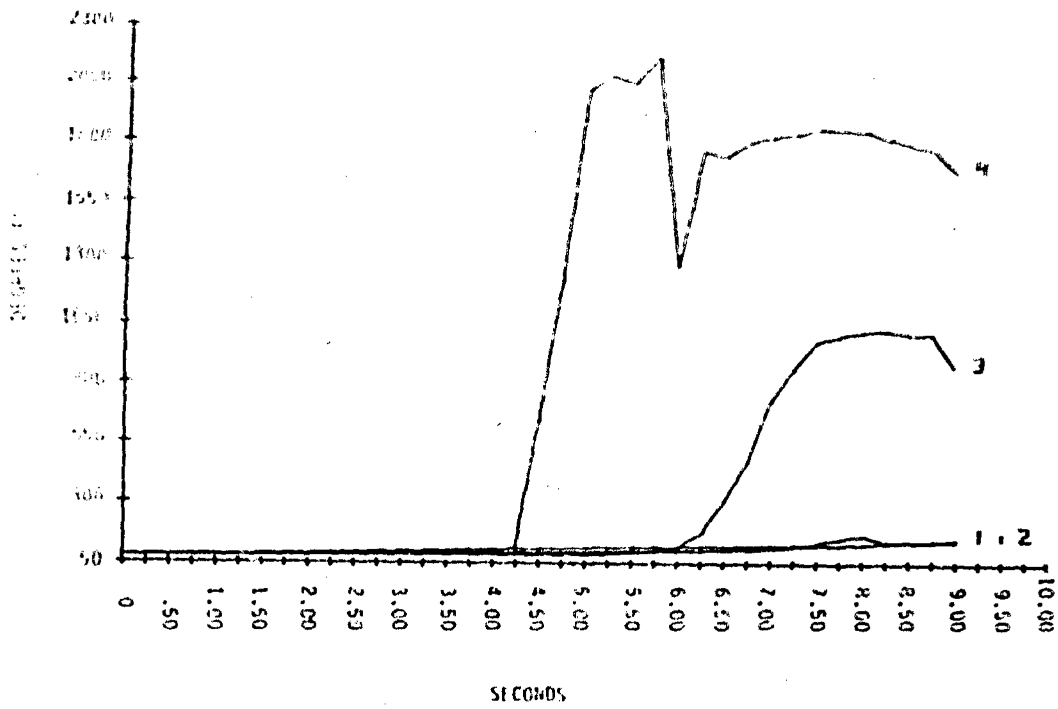

Figure 31. 30% Carbon Black Sample, Temperature-vs-Time Trace.

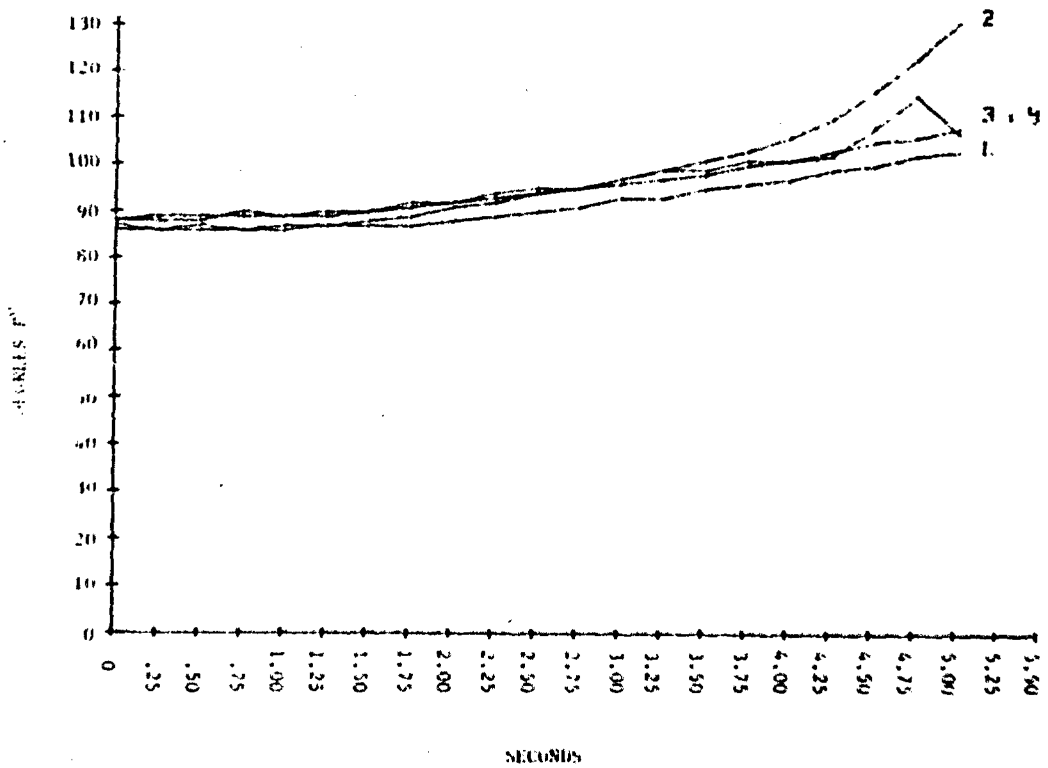

Figure 32. 25% Carbon Black, LiF, CAO-14 Sample, Temperature-vs-Time Trace.

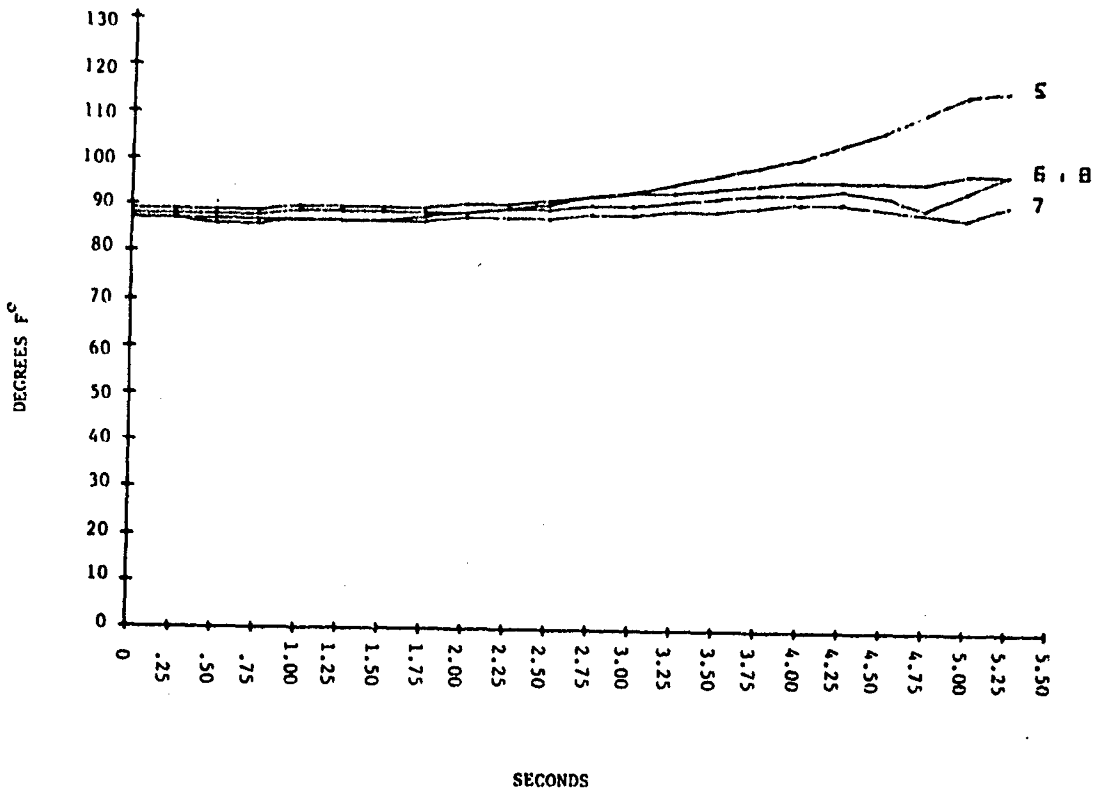

Figure 33. 20% Carbon Black, LiF, CAO-14 Sample, Temperature-vs-Time Trace.

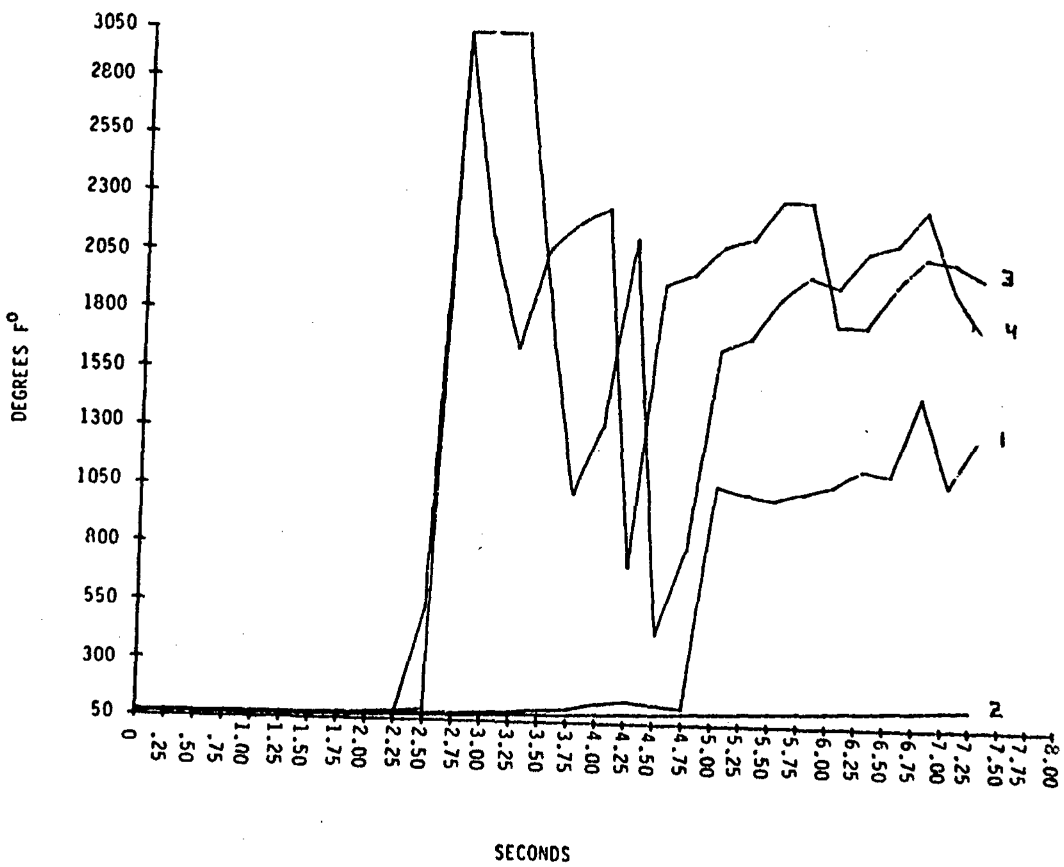

Figure 34. 90% Glass Beads, LiF, CAO-14 Sample, Temperature-vs-Time Trace.

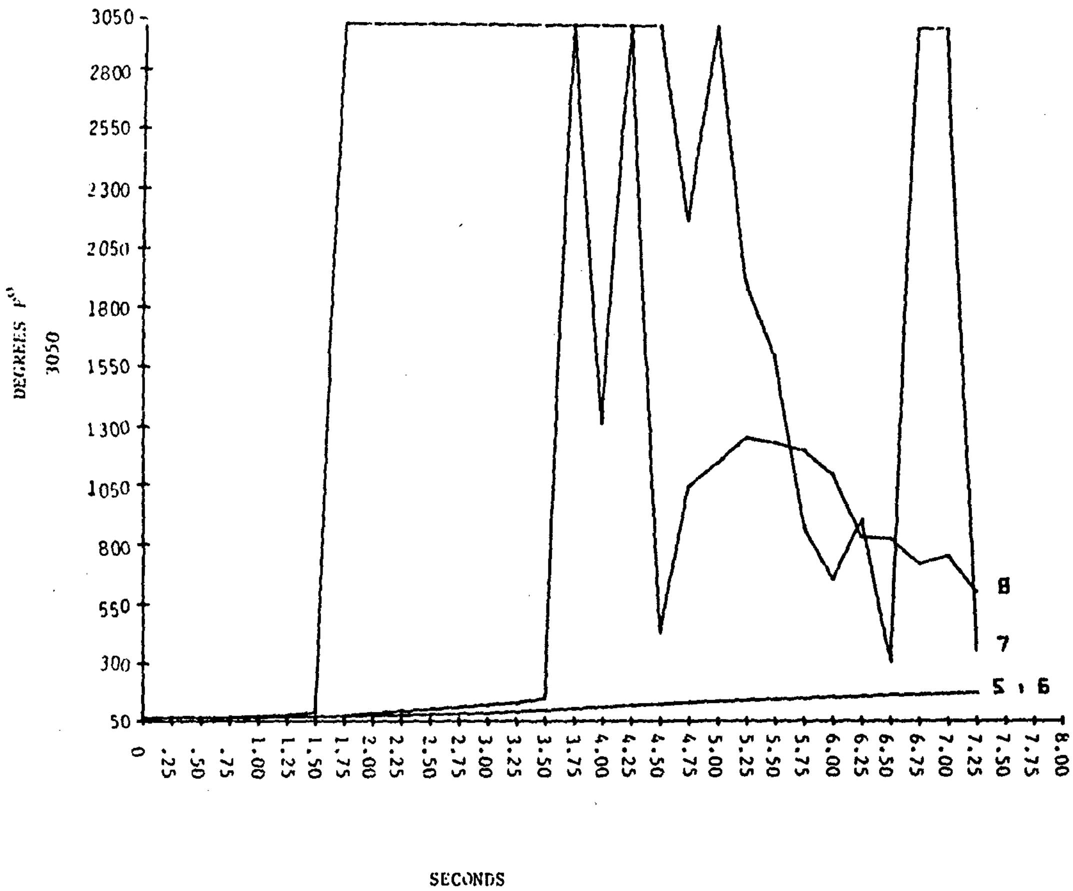

Figure 35. 90% Sand Sample, Temperature-vs-Time Trace.

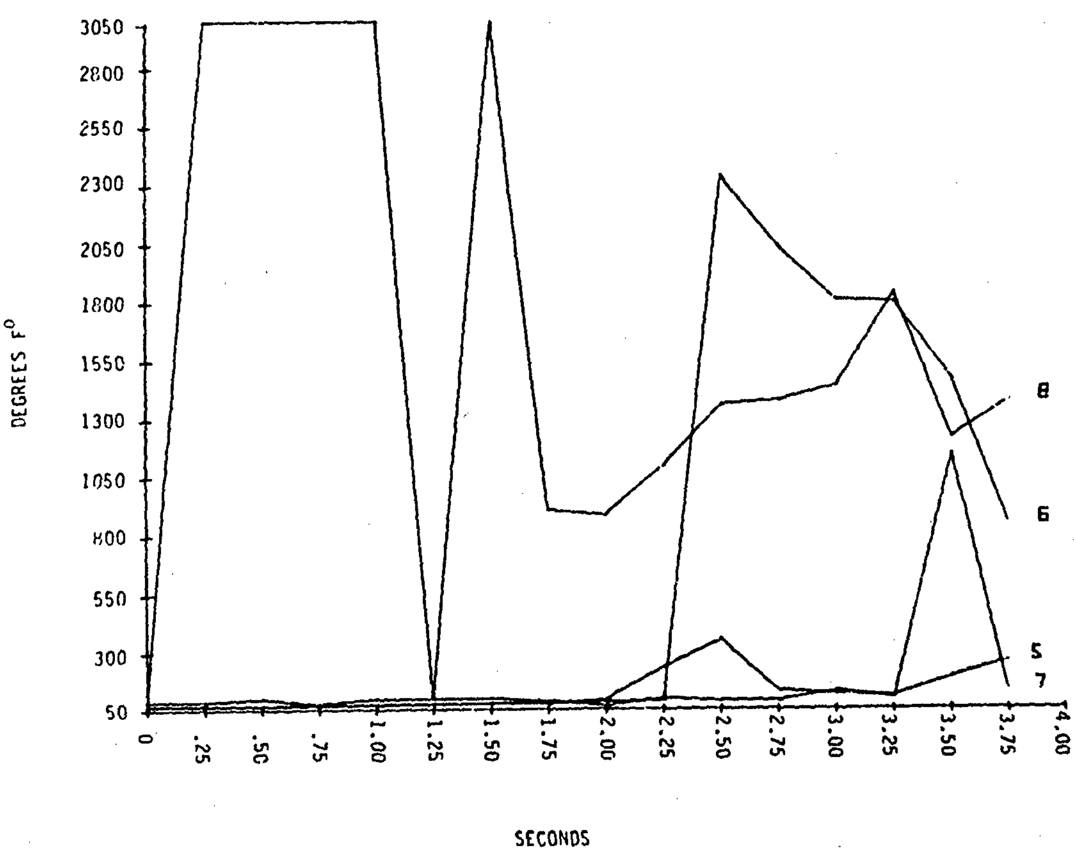

Figure 36. 25% Carbon Black Sample, Temperature-vs-Time Trace.

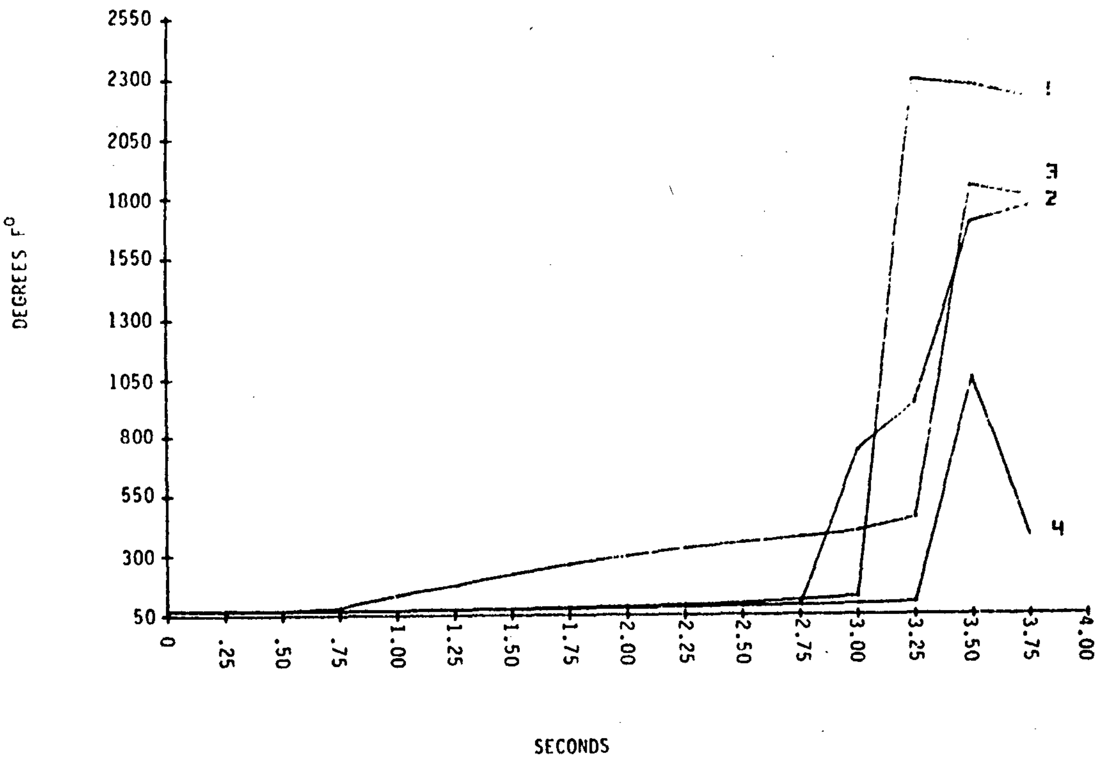

Figure 37. 80% Sand, LiF, CAO-14 Sample, Temperature-vs-Time Trace.

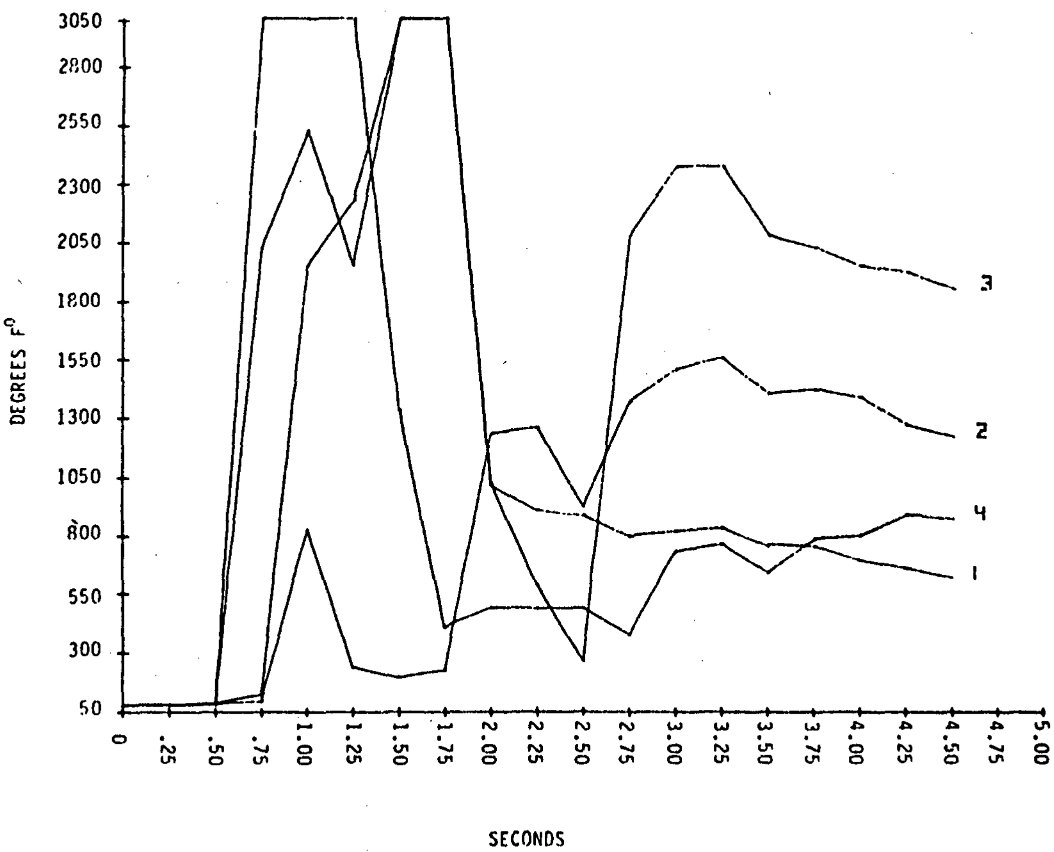

Figure 38. 35% Carbon Black Sample, Temperature-vs-Time Trace.

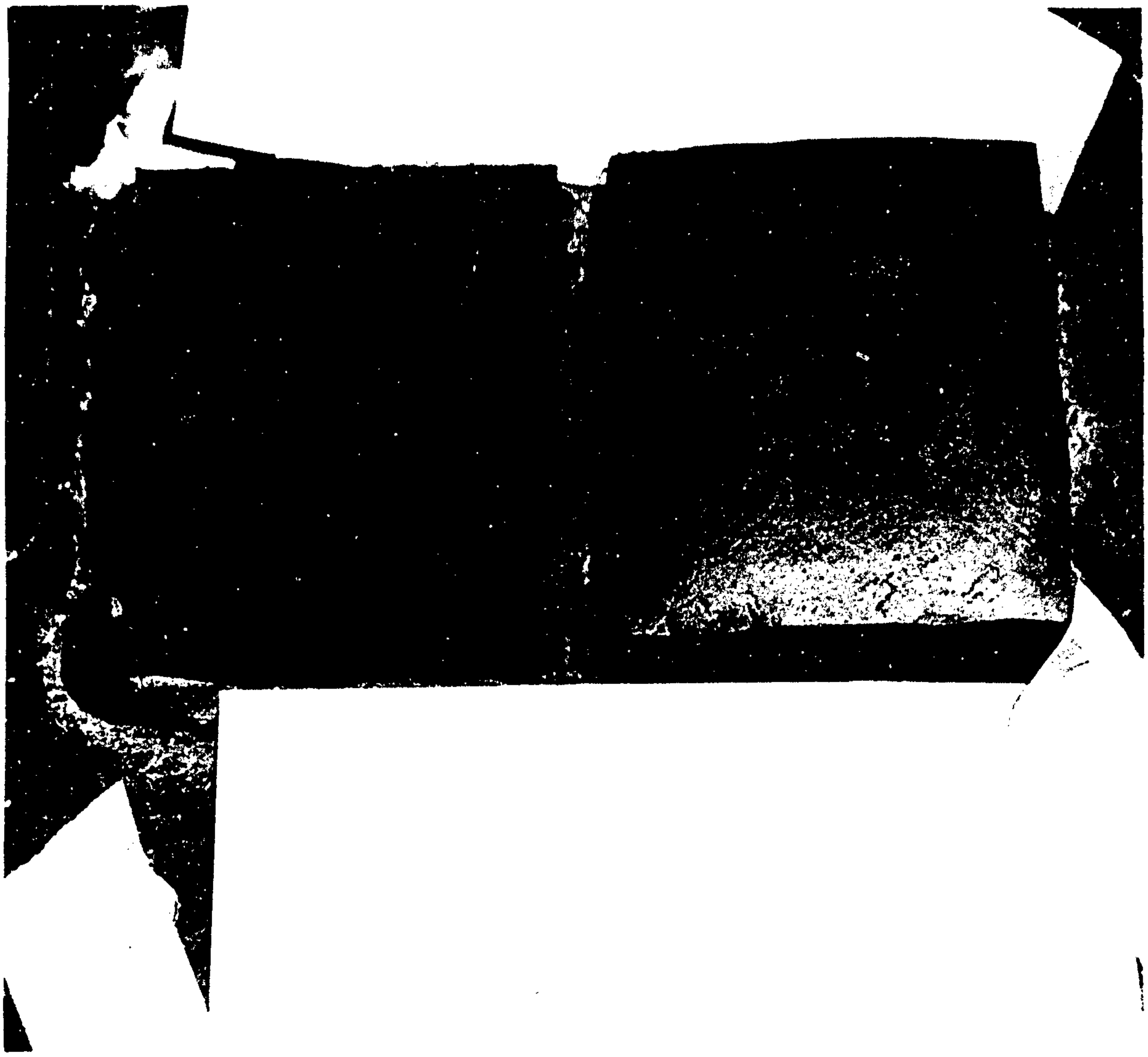

Figure 39. 80% Sand and 80% Graphite Sample, Pre Photograph.

Figure 40. 80% Sand and 80% Graphite Sample, Post Photograph.

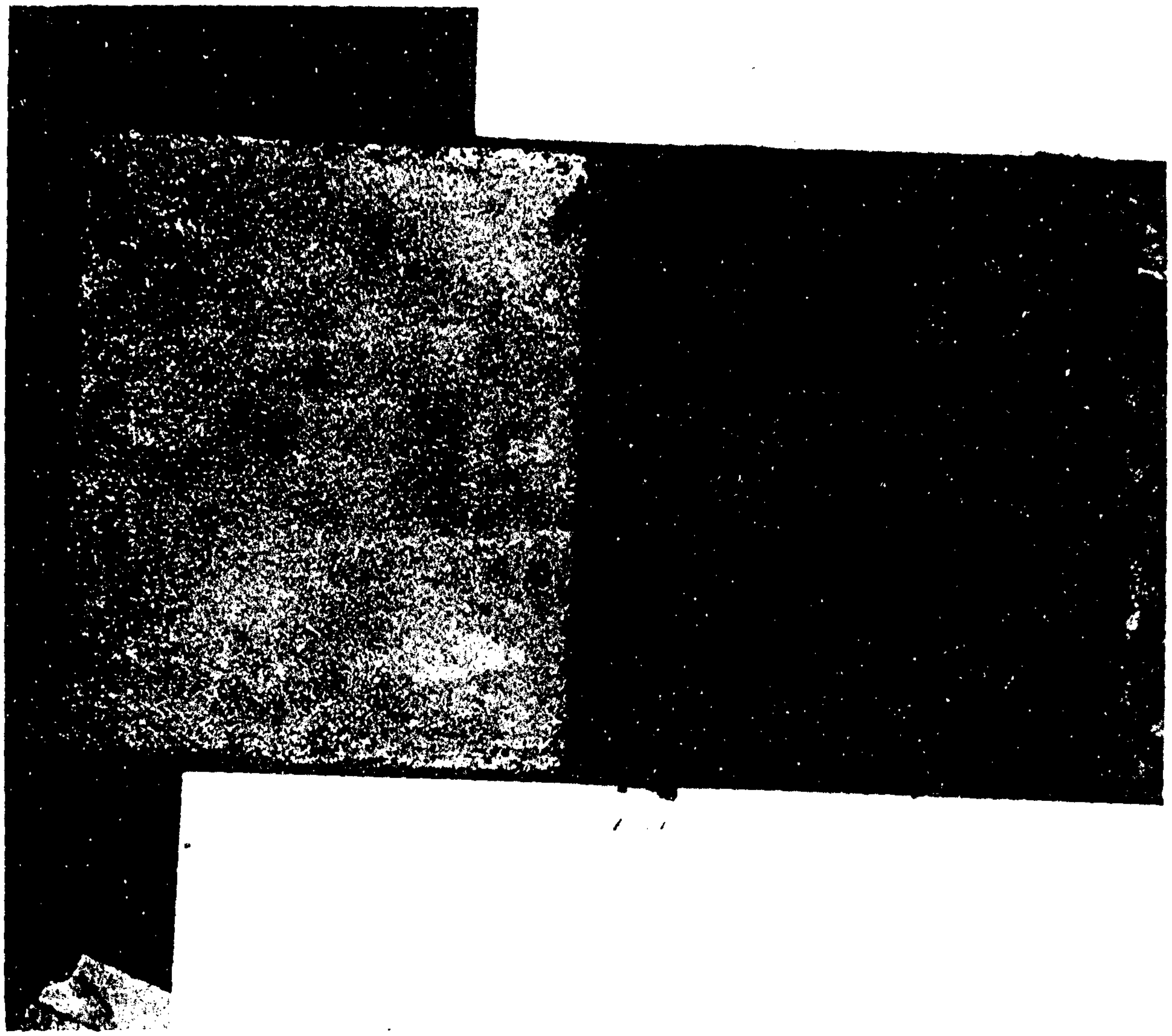

Figure 41. 80% Graphite and 90% Glass Beads Sample, Pre Photograph.

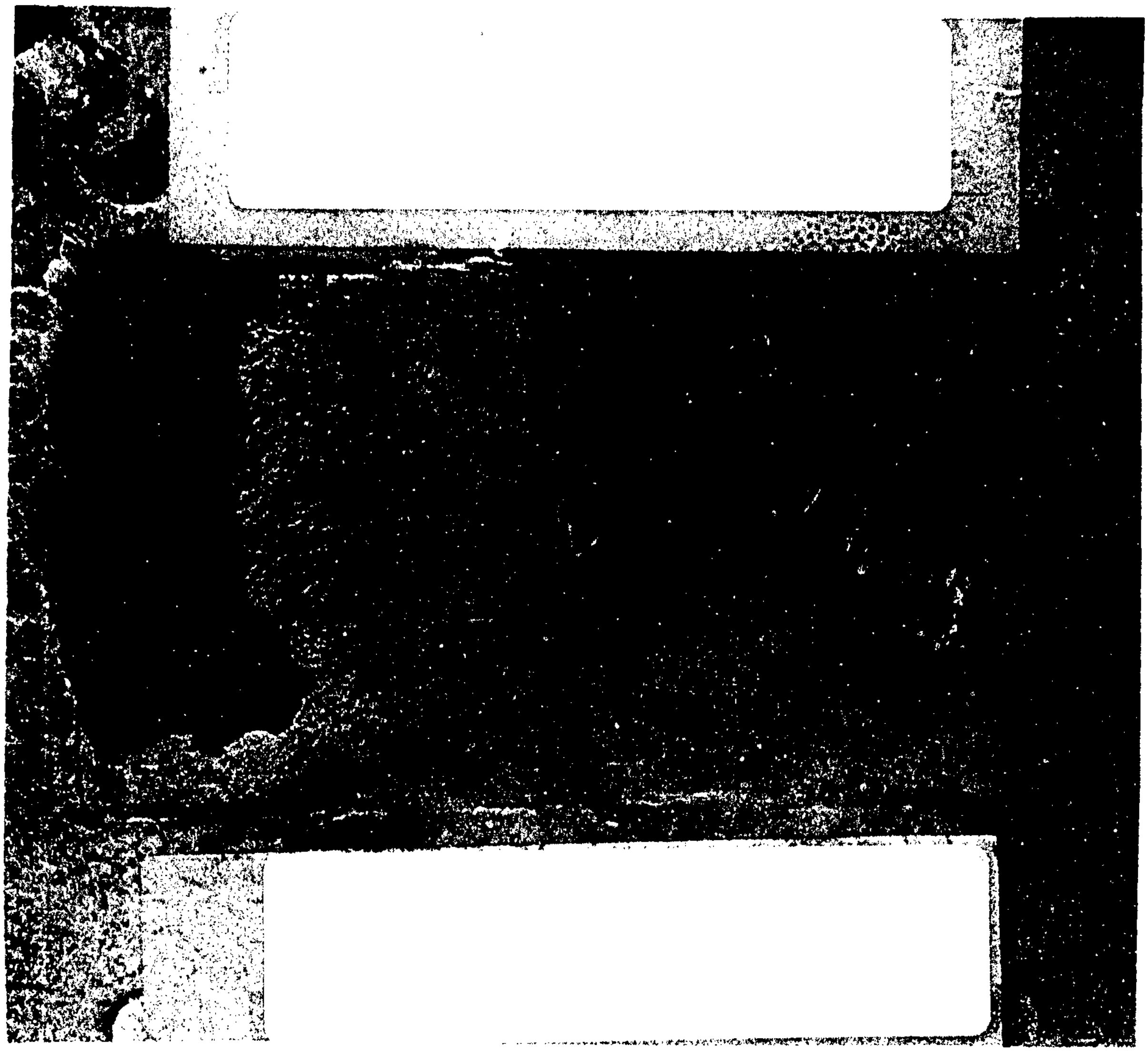

Figure 42. 80% Graphite and 90% Glass Beads Sample, Post Photograph.

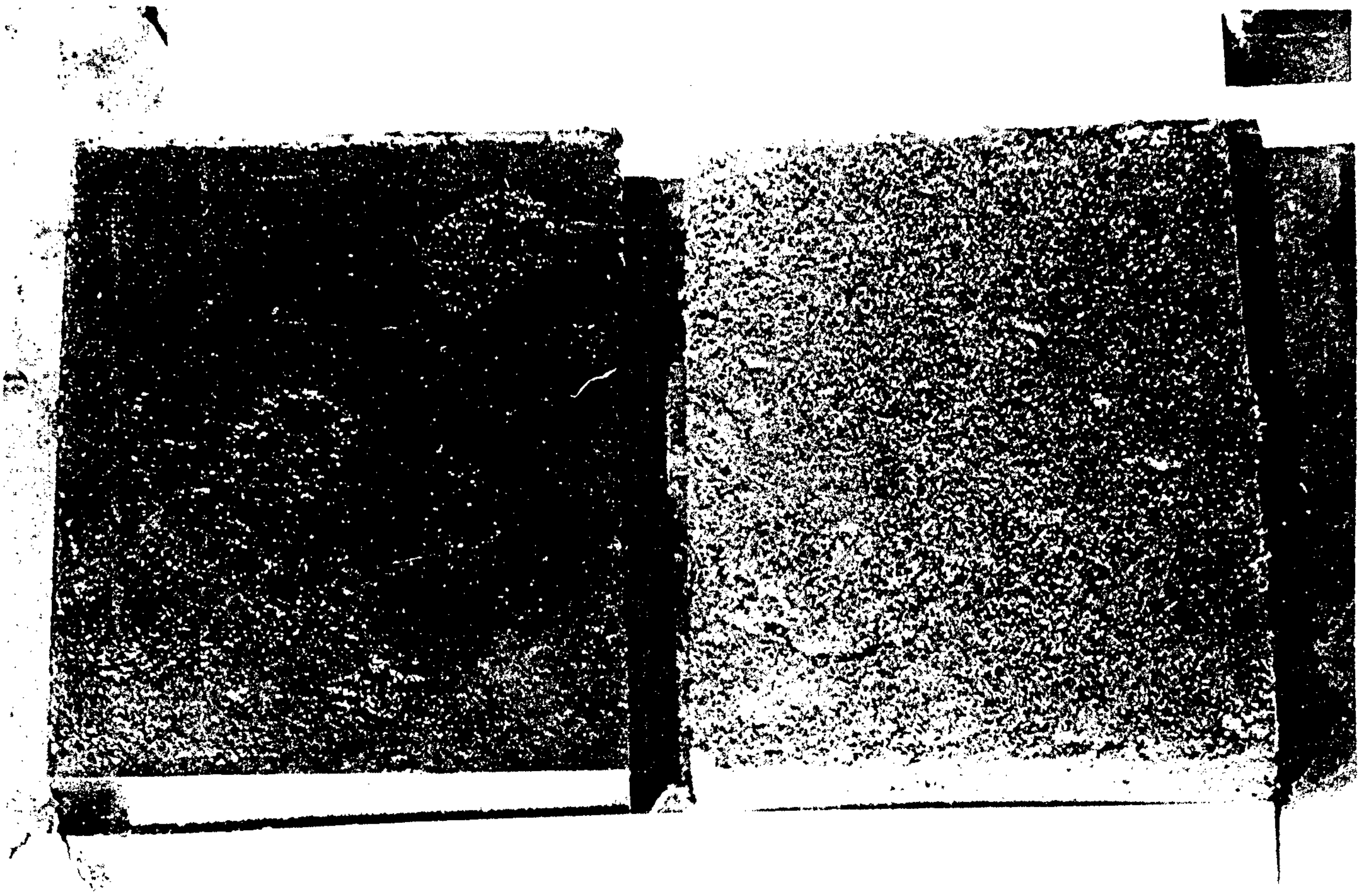

Figure 43. 80% Sand and 30% Carbon Black Sample, Pre Photograph.

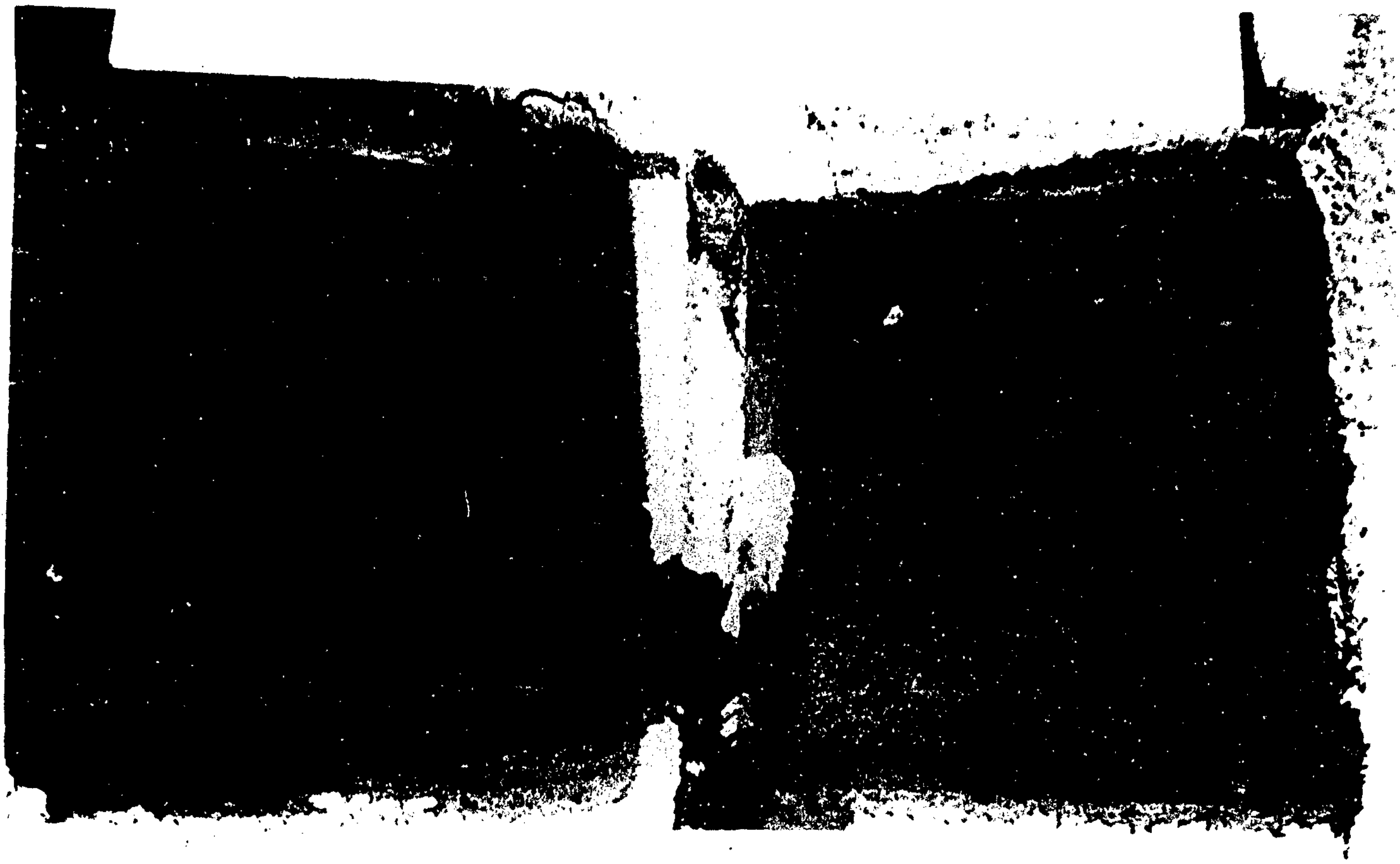

Figure 44. 80% Sand and 30% Carbon Black Sample, Post Photograph.

Figure 45. 25% Carbon Black and 20% Carbon Black Sample, Pre Photograph.

Figure 46. 25% Carbon Black and 20% Carbon Black Sample, Post Photograph.

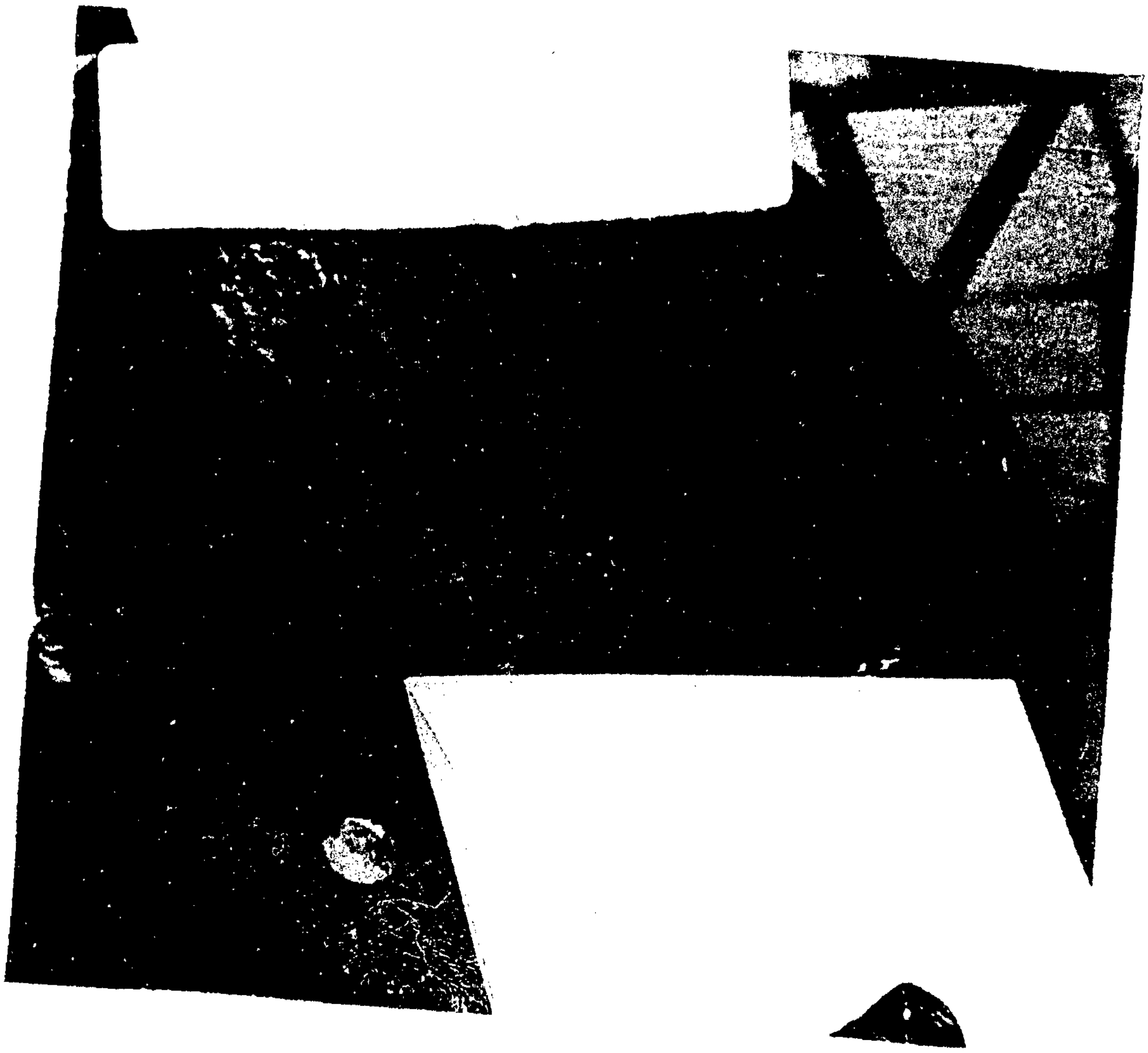

Figure 47. 90% Glass Beads and 90% Sand Sample, Pre Photograph.

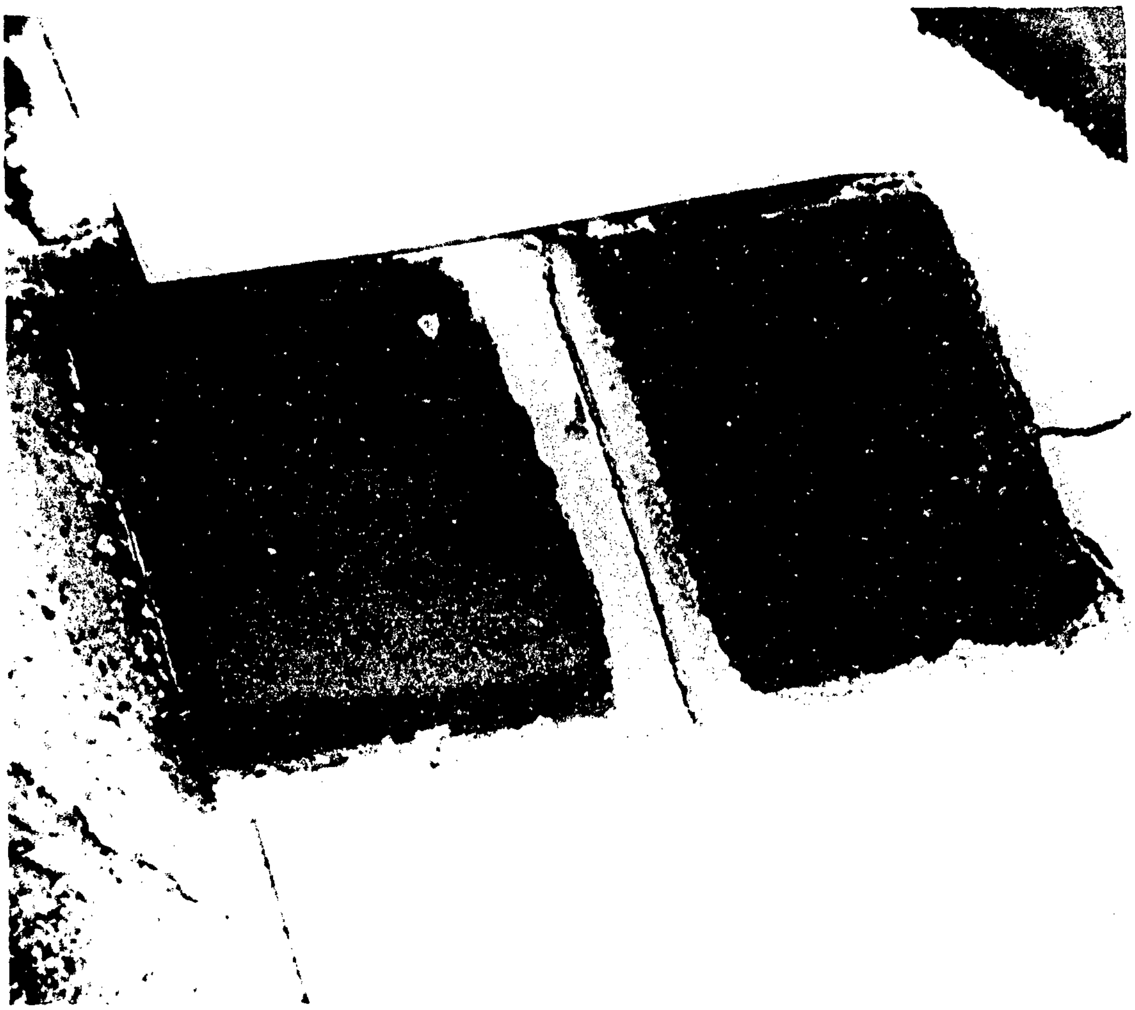

Figure 48. 90% Glass Beads and 90% Sand Sample, Post Photograph.

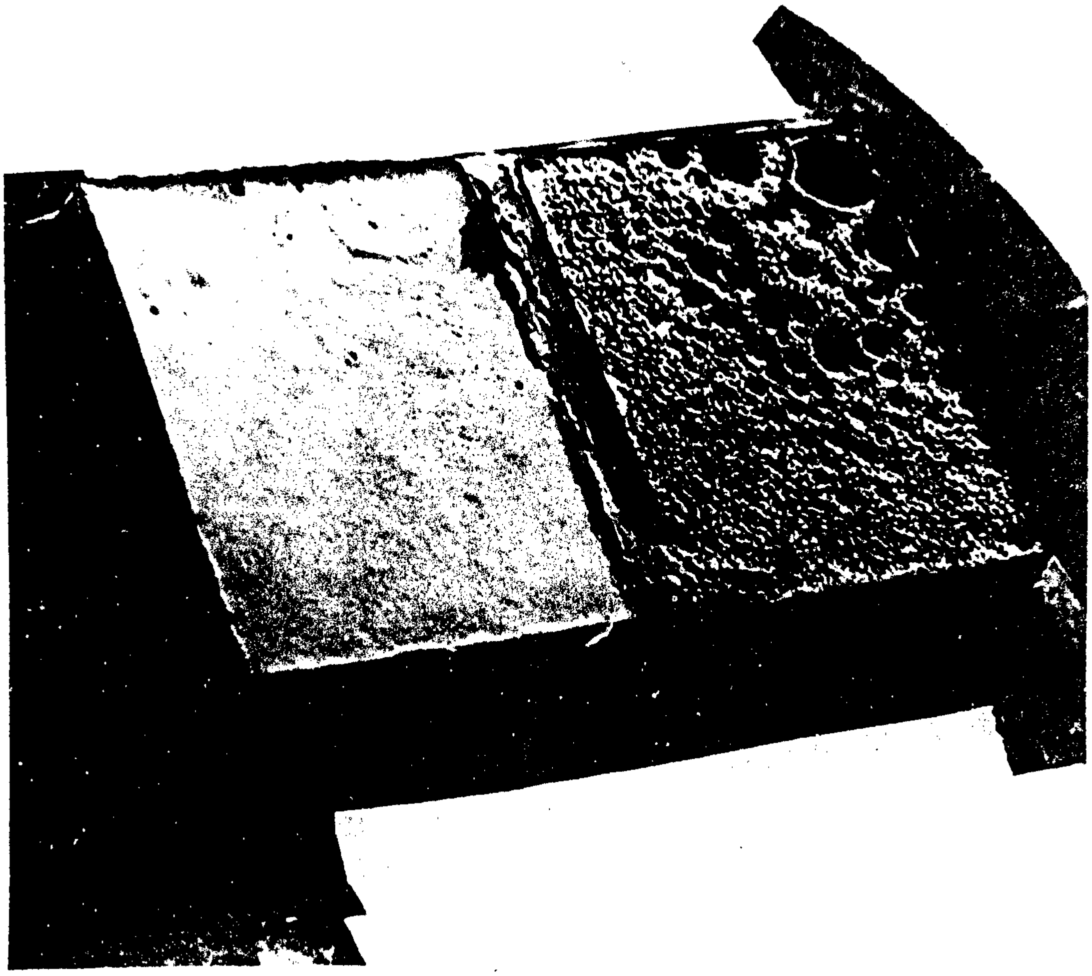

Figure 49. 80% Sand and 25% Carbon Black Sample, Pre Photograph.

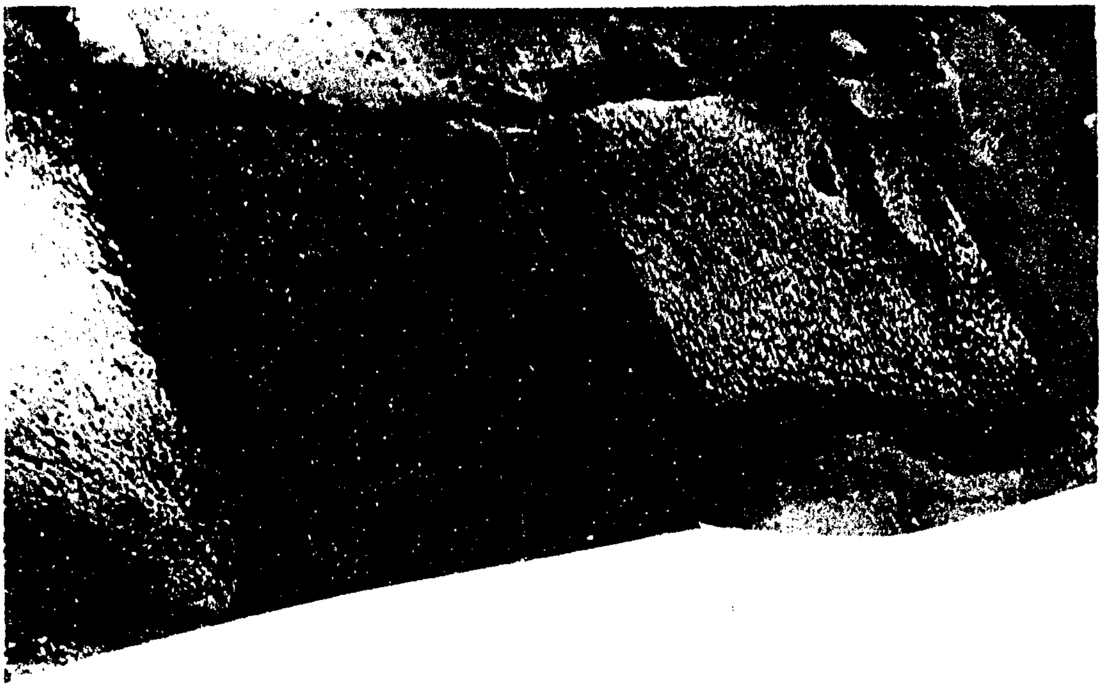

Figure 50. 80% Sand and 25% Carbon Black Sample, Post Photograph.

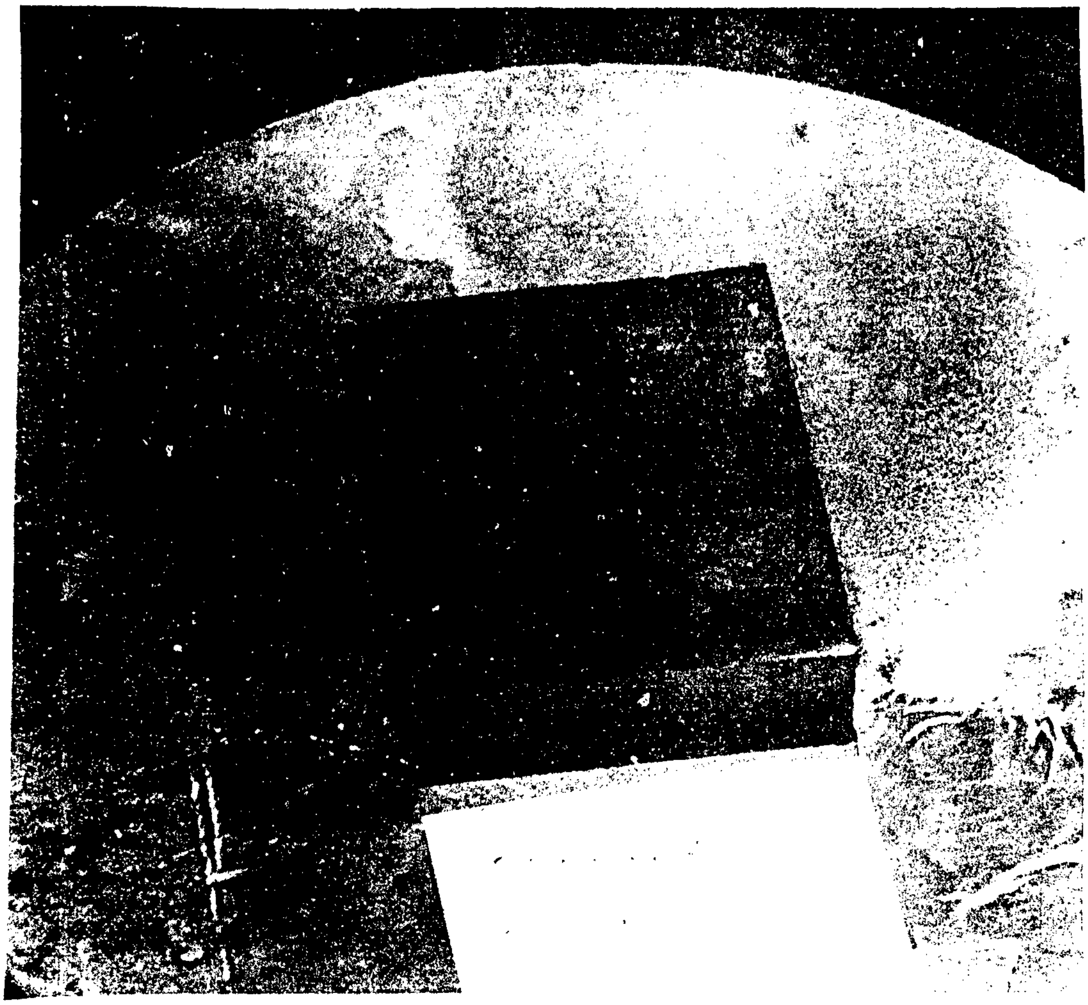

Figure 51. 35% Carbon Black Sample, Pre Photograph.

Figure 52. 35% Carbon Black Sample, Post Photograph.

3.3. Large Motor Firings.

The objective of Phase III was to scale up selected formulations from Phase II to the one-gallon size. These scaled up mixes were subjected to more rigorous experiments, such as the exhaust of the actual field tests of the Flight Termination Ordnance System (FTOS) of the Peacekeeper Stages I, II, and III performed at the AFRPL; the AFRPL's firing of a short-length, super HIPPO and the Kennedy Space Center's STS-5 launch of the Space Shuttle, Columbia. The following paragraphs describe each firing.

3.3.1. Peacekeeper Stage III Experiments.

Three one-gallon mixes were made, using the same formulation of 90% sand without a flame suppressant or antioxidant. Two of the one-gallon mixes were cast in blocks 3 1/2 inches thick, 8 inches wide, and 10 inches long (see Figure 53). Both samples cured rock-hard in a matter of hours. The samples were placed 15 feet from the nozzle in direct line with the exhaust. The third one-gallon mix was cast on one of the rear support beams. This sample was 2 inches thick, 8 inches wide, and 18 inches long (see Figure 54). This sample was compared with Dow-Corning's DC 93-058 insulating product that was also applied to the rear support beams.

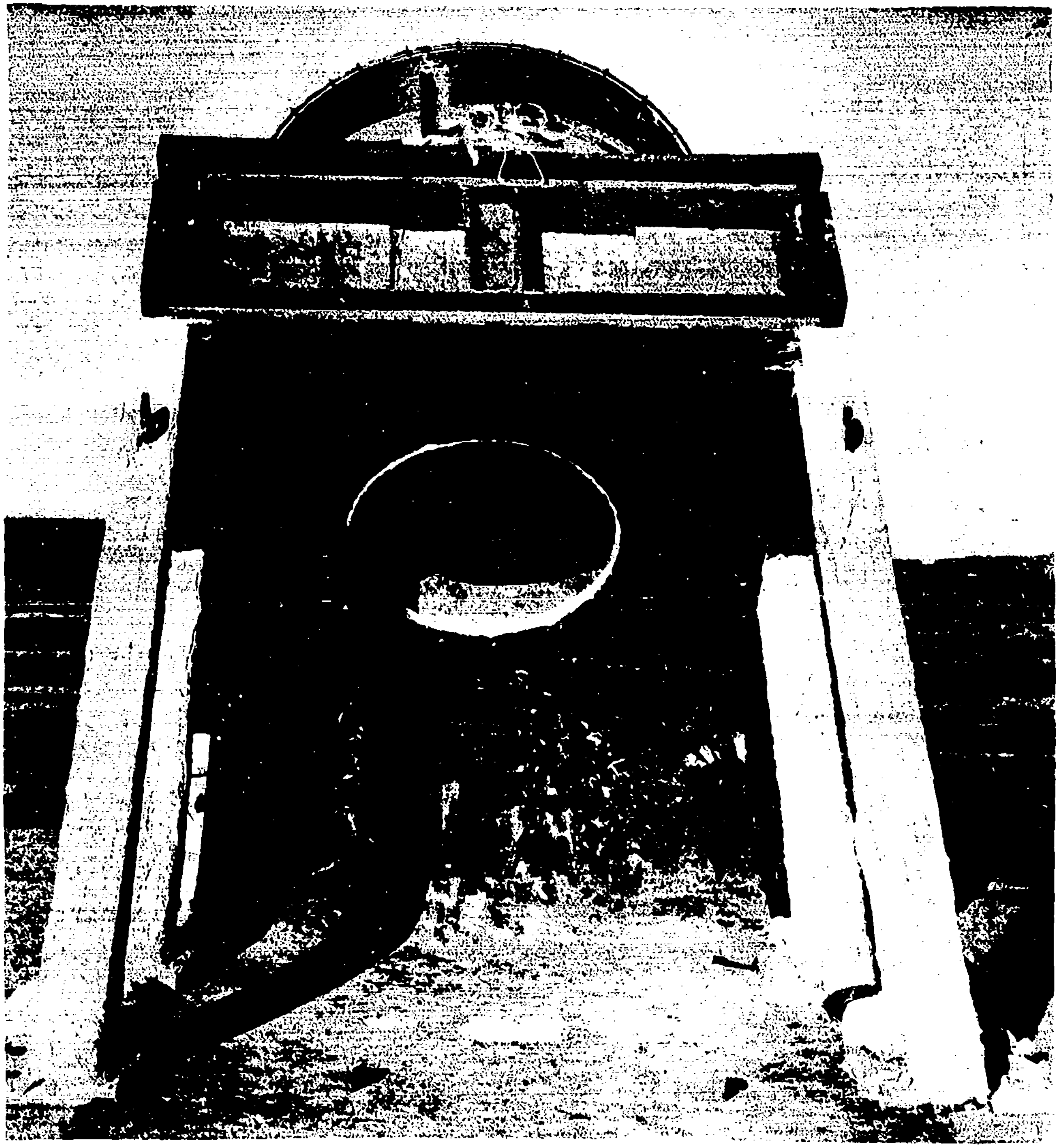

Figure 53. Peacekeeper Stage III FTOS.

Figure 54. Peacekeeper Stage III Support Beam Sand Sample.

The firing lasted 2.7 seconds before FTOS initiated. One of the samples placed on the flame deflector eroded less than 1/2 inch at the leading edge (see Figure 55). The other sample was blown away and found in two pieces (see Figure 56). The sample on the rear support beam survived the experiment better than the Dow-Corning product (see Figure 57). No erosion was evident on our product; however, the DC 93-058 debonded in several areas (see Figure 57). The beams incurred no damage, but the zinc paint was burned.

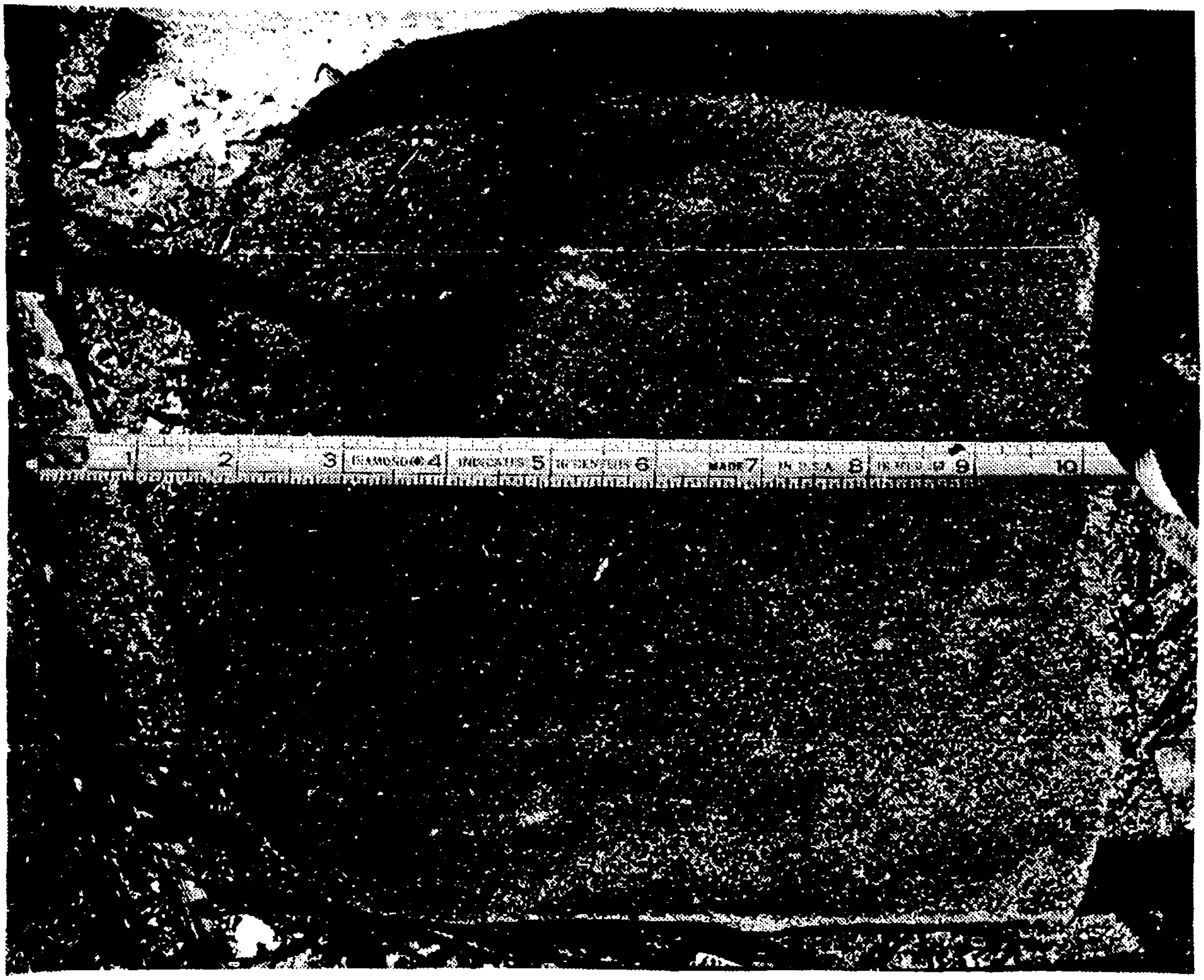

Figure 55. Post Photograph of Sand Samples on Flame Trench.

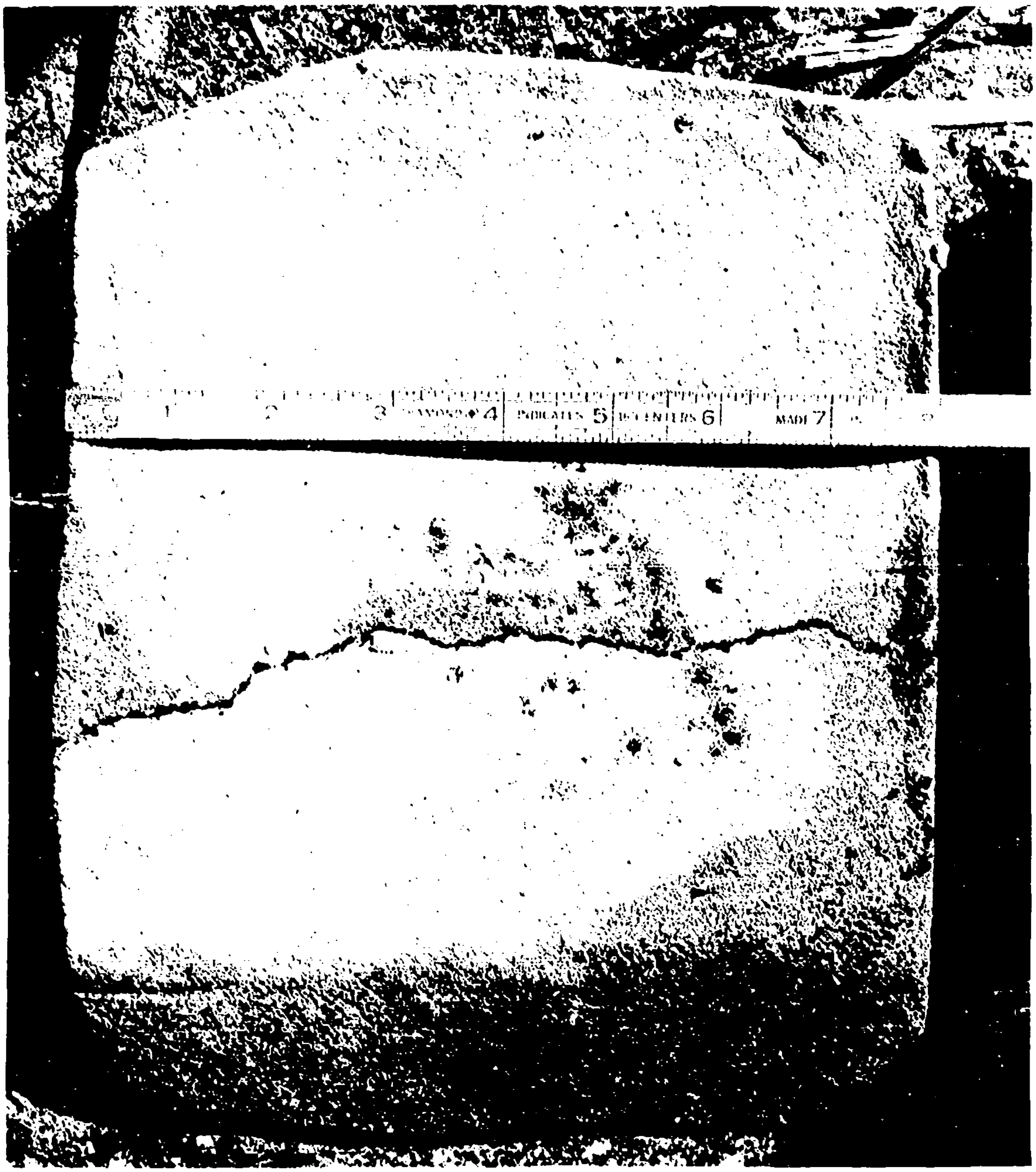

Figure 56. Post Photograph of Sand Sample Broken in Two.

Figure 57. Post Photograph of Debonding of DC 93-058.

3.3.2. Peacekeeper Stage I Experiments.

The samples were placed at different distances from the motor to determine the thickness of the material that would provide the best protection. The leading edges of the samples were cut at different angles to help determine the thickness required for the insulation to perform at its best.

The firing lasted 2.5 seconds before FTOS initiated. All of the samples survived, and they protected the concrete under them, even though the concrete around the samples melted and bubbled due to the extreme heat (see Figure 58 for reference of the sample to the motor). Table 3 contains the post-measurements of the samples. The optimum thickness referenced in Table 3 is one inch. As the duration of the test increases we suggest the thickness of the material be increased in the most critical areas.

Table 3. Results of Samples, Peacekeeper Stage I.

Sample #	Thickness (in)	Width (in)	Length (in)
1	3/6	2 3/4	2 1/2
2	1/2	4	4 1/2
3	3/4	4	5
4	3/16	4	5
5	1/4	2 1/2	5
6	1/2	4	5
7	1/2	4	5
8	1/2	4	5
9	1/2	4	5

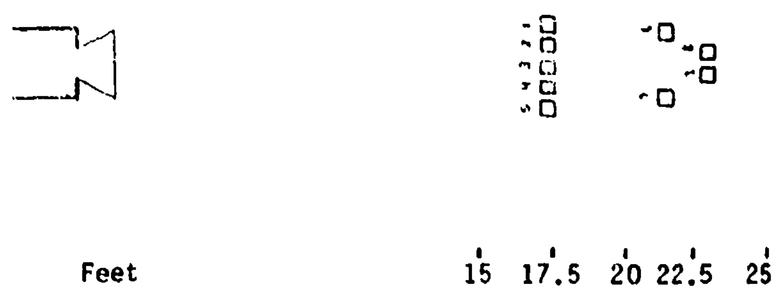

Figure 58. Placement and Alignment of Samples on Peacekeeper Stage I.

3.3.3. Peacekeeper Stage II Experiments.

In this experiment, nine samples composed of carbon black, glass beads, and sand were tested. They were cast in the same dimensions as used in the Peacekeeper Stage I experiment. Figures 59 and 60 show the placement and alignment of the samples before the firing. In Figure 60, the dark square areas are the previous Peacekeeper FTOS sample locations. This firing lasted 0.3 seconds before FTOS initiated.

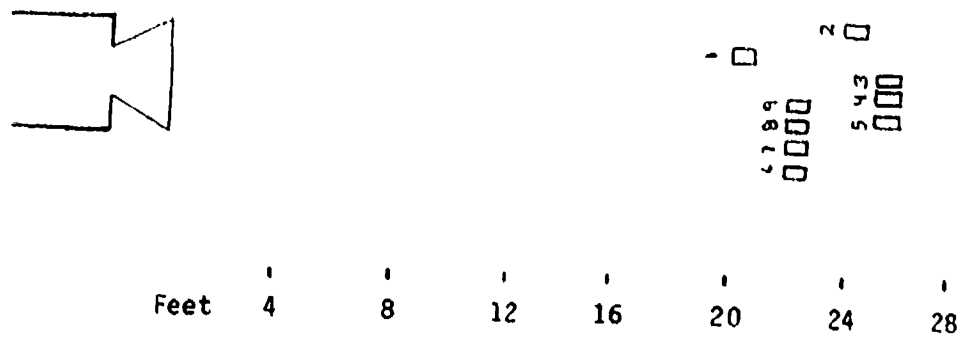

Figure 59. Placement and Alignment of Samples on Peacekeeper Stage II.

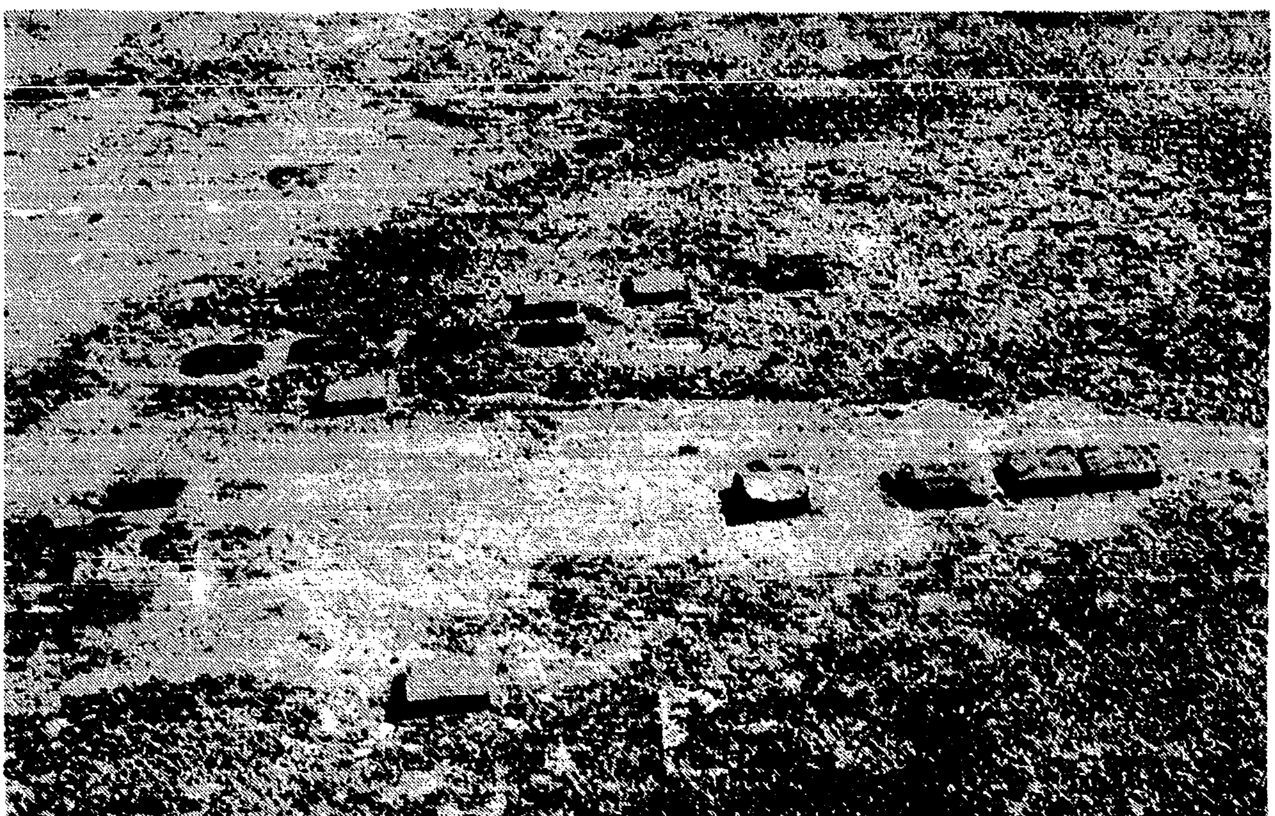

Figure 60. Placement of Samples, Peacekeeper Stage II.

All of the samples survived (see Figure 61). Two of the three carbon black samples came loose due to the force of the ejecting debris, which was produced by the motor when FTOS was initiated. The results of this firing are in Table 4.

Table 4. Results of Samples, Peacekeeper Stage II.

Sample	Solids Used	Results
1	80% SiO_2	Eroded 1/4 inch.
2	80% SiO_2	No erosion.
3	80% SiO_2	Eroded 1/2 inch.
4	80% SiO_2	Eroded 1/4 inch.
5	90% Carbon Black	Eroded 1/2 inch.
6	80% Carbon Black	Lost, due to force of debris.
7	90% Carbon Black	Lost, due to force of debris.
8	90% Glass Beads	Eroded 3/8 inch.
9	90% Glass Beads	Eroded 1/2 inch.

Figure 61. Post-Results, Peacekeeper Stage II.

3.3.4. Super HIPPO Experiments.

Three one-gallon mixes of carbon black were exposed to the radiant heat produced by the short-length, super HIPPO rocket motor. Thermocouples were placed about 2 inches apart, 12 inches from the exit cone, and at the same height as the exit cone. The thickness of the samples covering each thermocouple was varied. Thicknesses were 1/4 inch, 1/2 inch, 1 inch, and 2 inches (see Figure 62). A carbon black patch about

1/4 inch thick was cast over Dow-Corning's DC 93-C58 material and placed next to the exit cone to achieve maximum concentration of radiant heat (see Figure 63). Dow-Corning's material was the principal insulation material used to protect the vital areas of the nozzle assembly, just as it was used to protect the support beams on the Peacekeeper Stage II stand (see Figure 64). The duration of the firing lasted 60 seconds.

Figure 62. Carbon Black Sample on Super HIPPO Aft Closure.

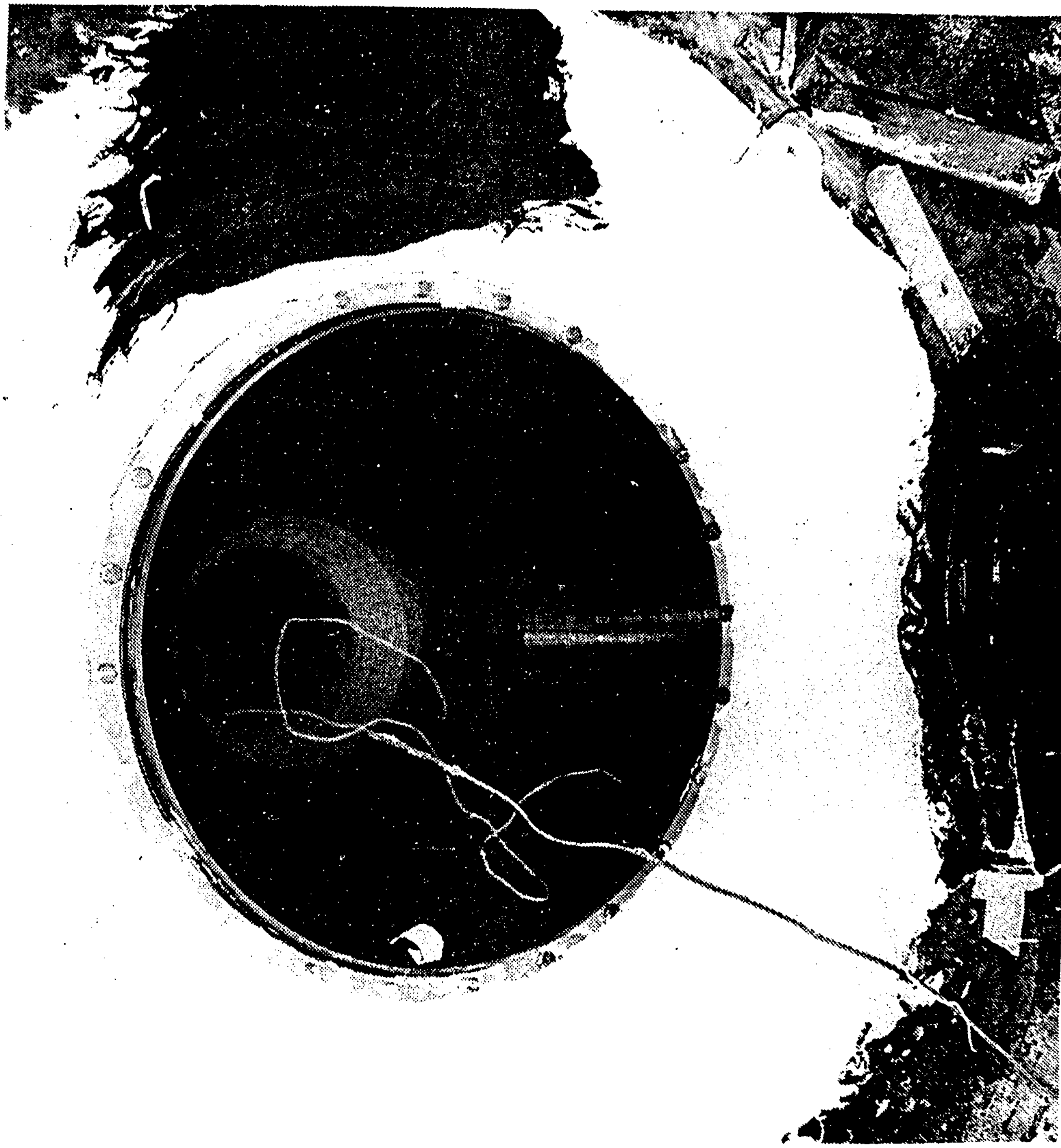

Figure 63. Carbon Black Sample over DC 93-058 near Exit Cone.

Figure 64. Overall View of Sample on Aft Closure, Super HIPPO.

The sample placed next to the exit cone on top of the Dow-Corning material burned away, but not before it protected the DC 93-058 (see Figures 65 and 66). The samples of various thicknesses that were placed on top of the closure also did very well (see Figures 67 and 68). Table 5 lists the erosion measurements of the samples. Figures 69 through 72 show the temperature-versus-time traces.

Table 5. Results of Samples, Super HIPPO.

Pre-Firing Measurement (in)	Post-Firing Measurement (in)
2	1 3/4
1	3/4
1/2	1/4
1/4	1/8

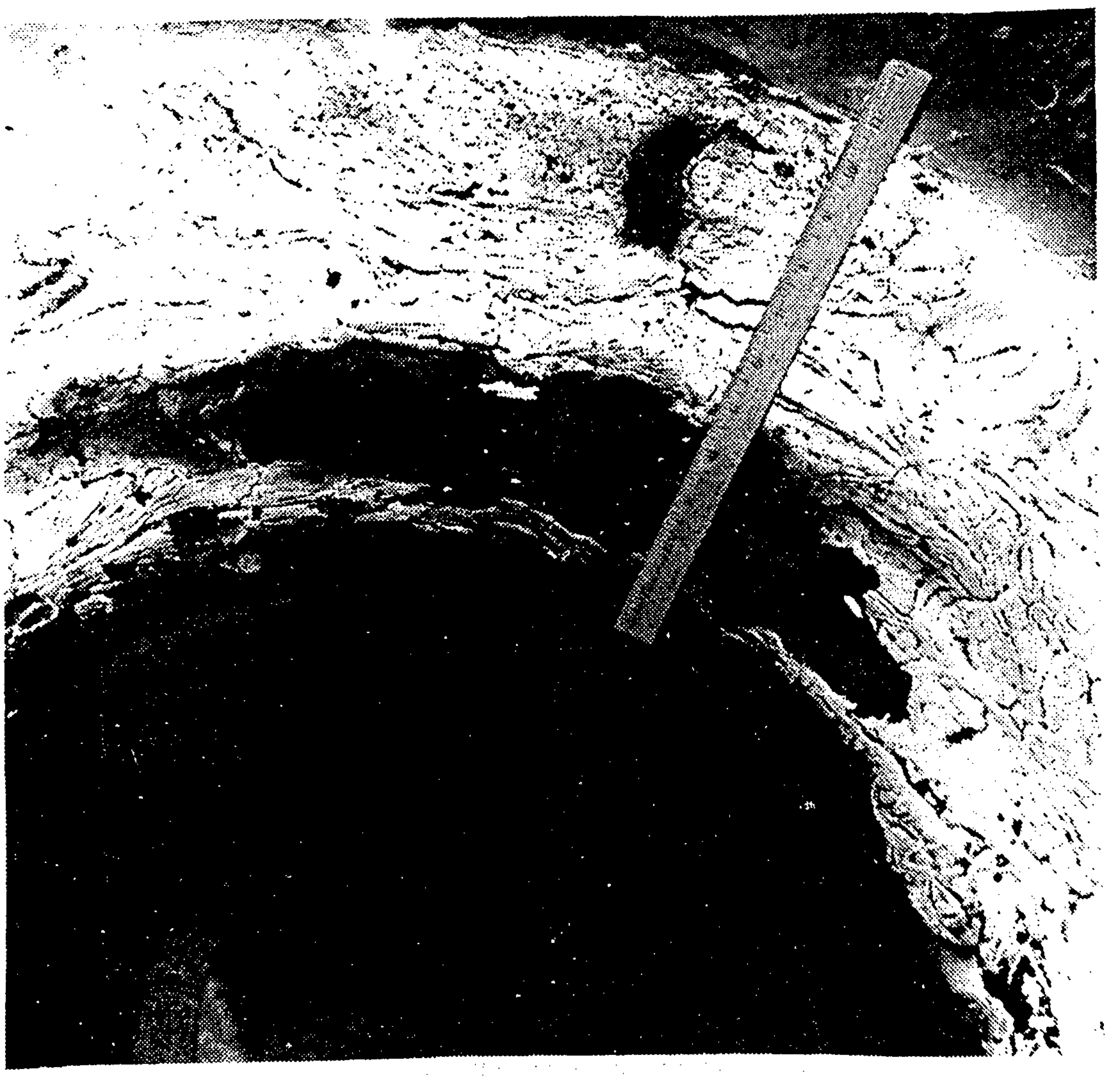

Figure 65. Post Result of Carbon Black over DC 93-058.

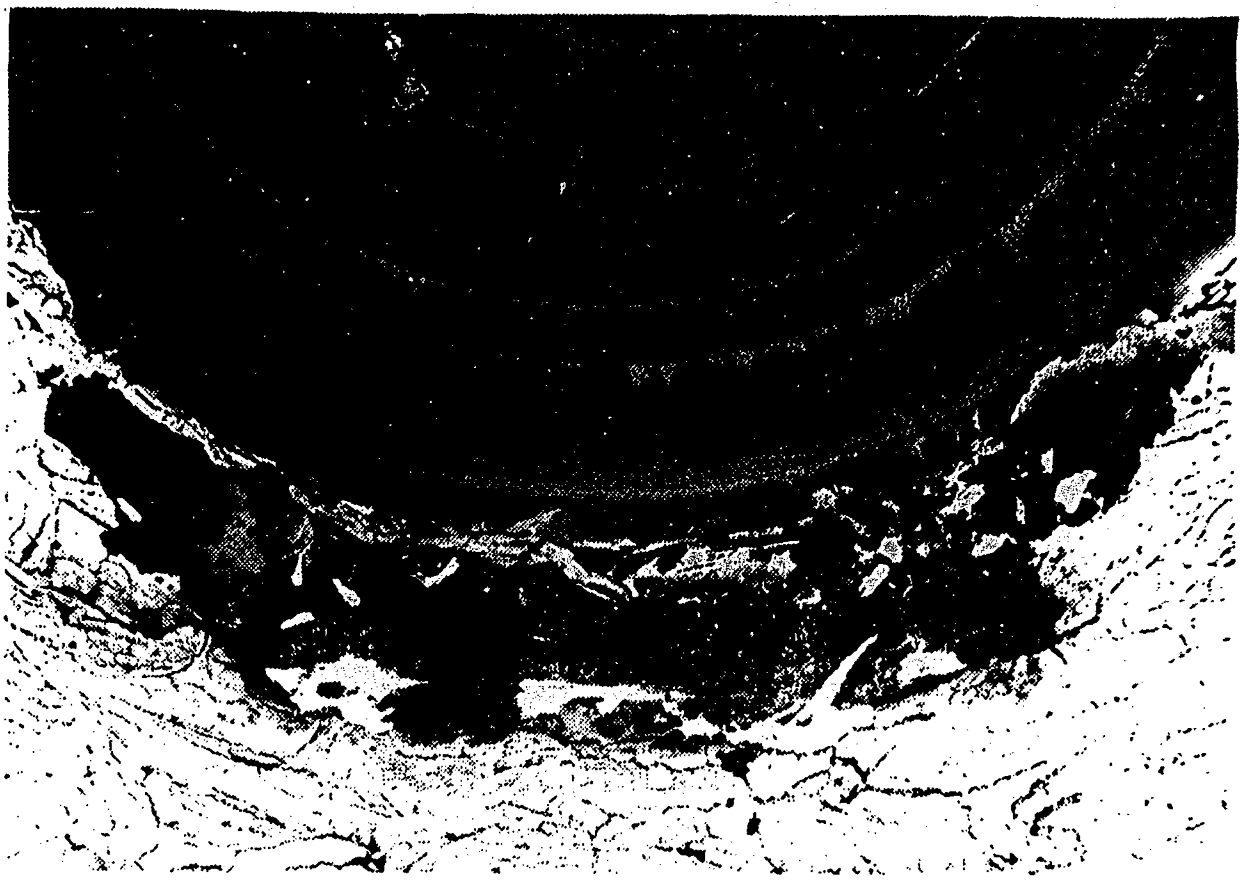

Figure 66. Close-up Post Result of Carbon Black over DC 93-058.

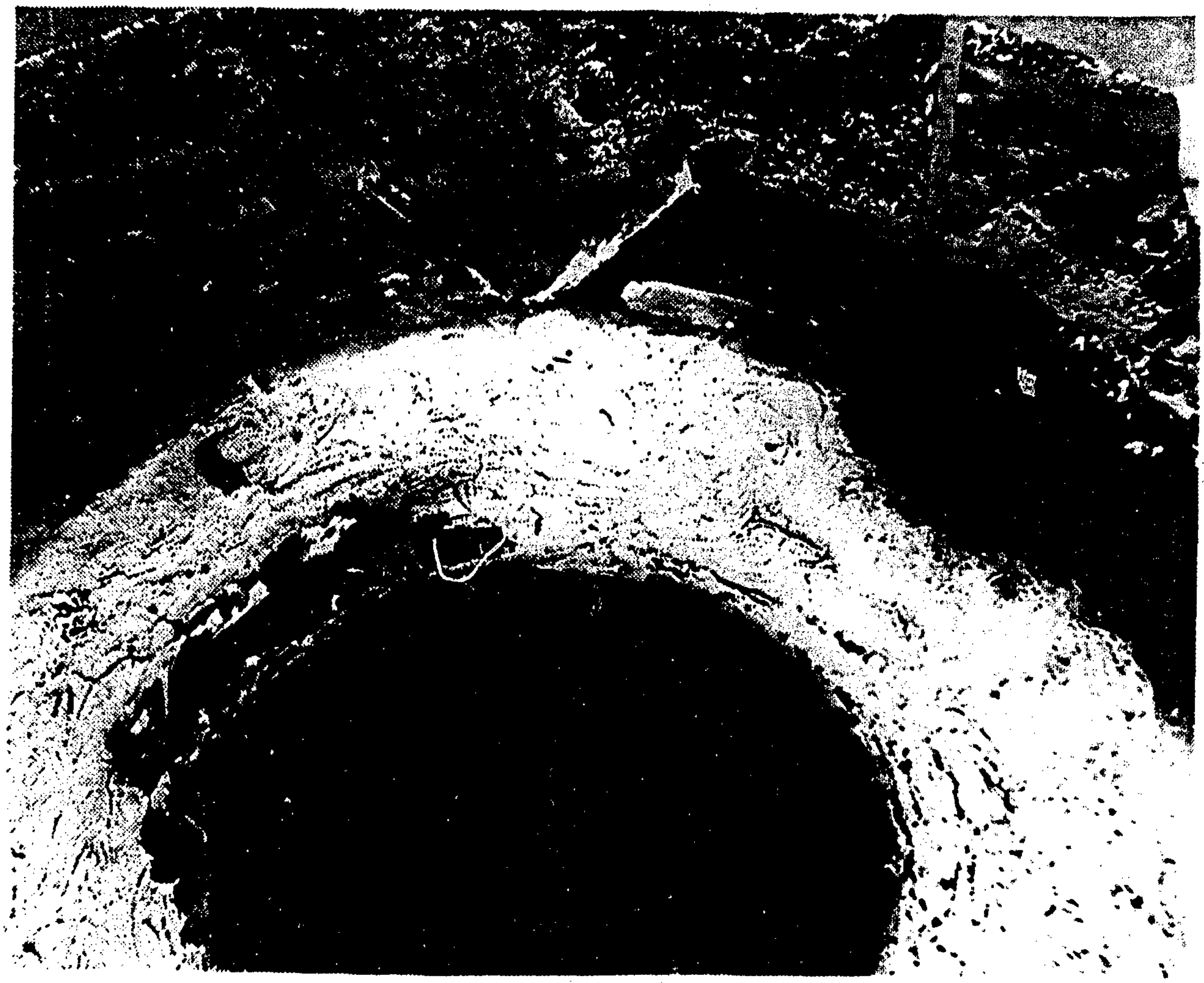

Figure 67. Post Result of Sample on Aft Closure, Super HIPPO.

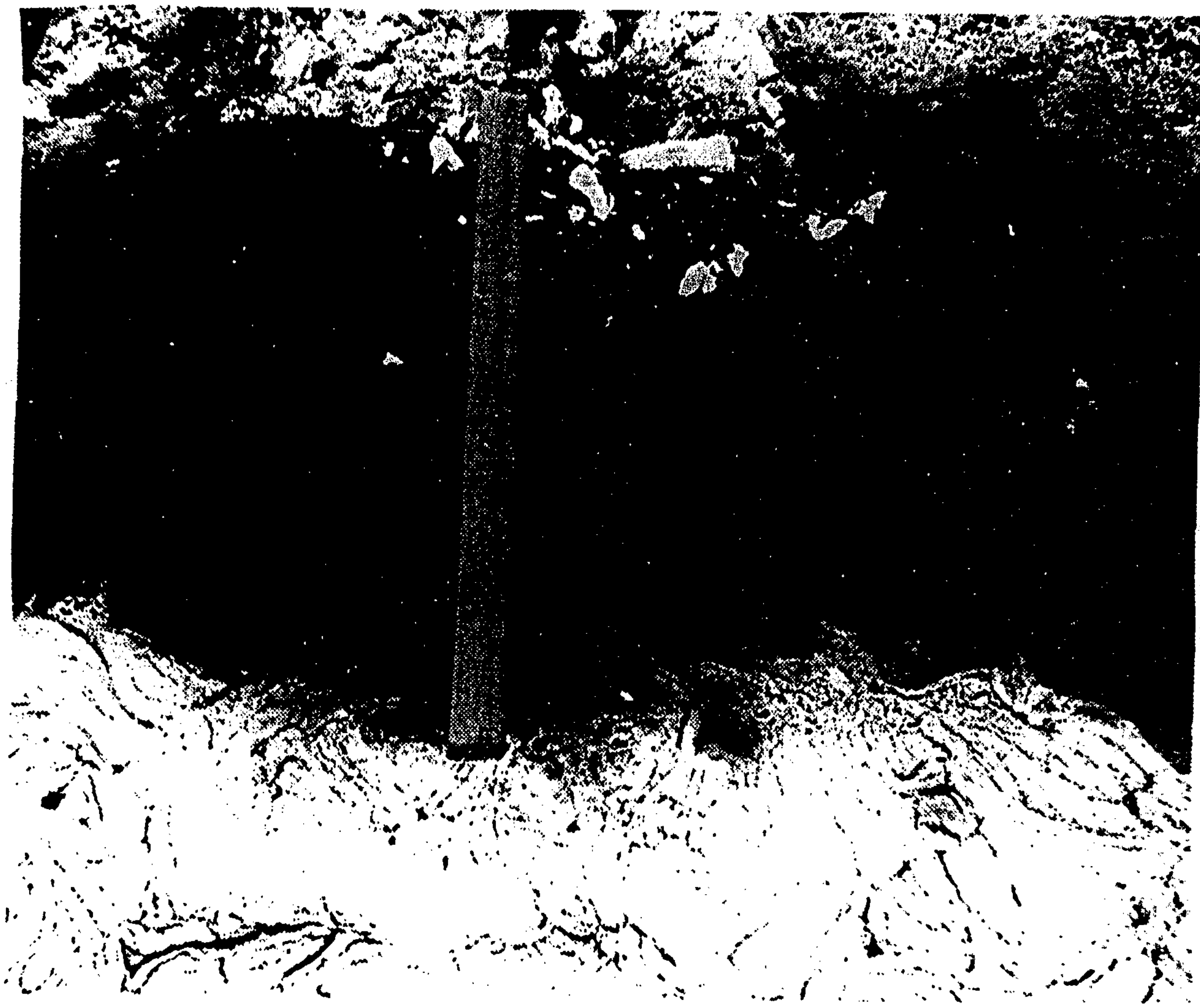

Figure 68. Close-up Post Result of Sample on Aft Closure, Super HIPPO.

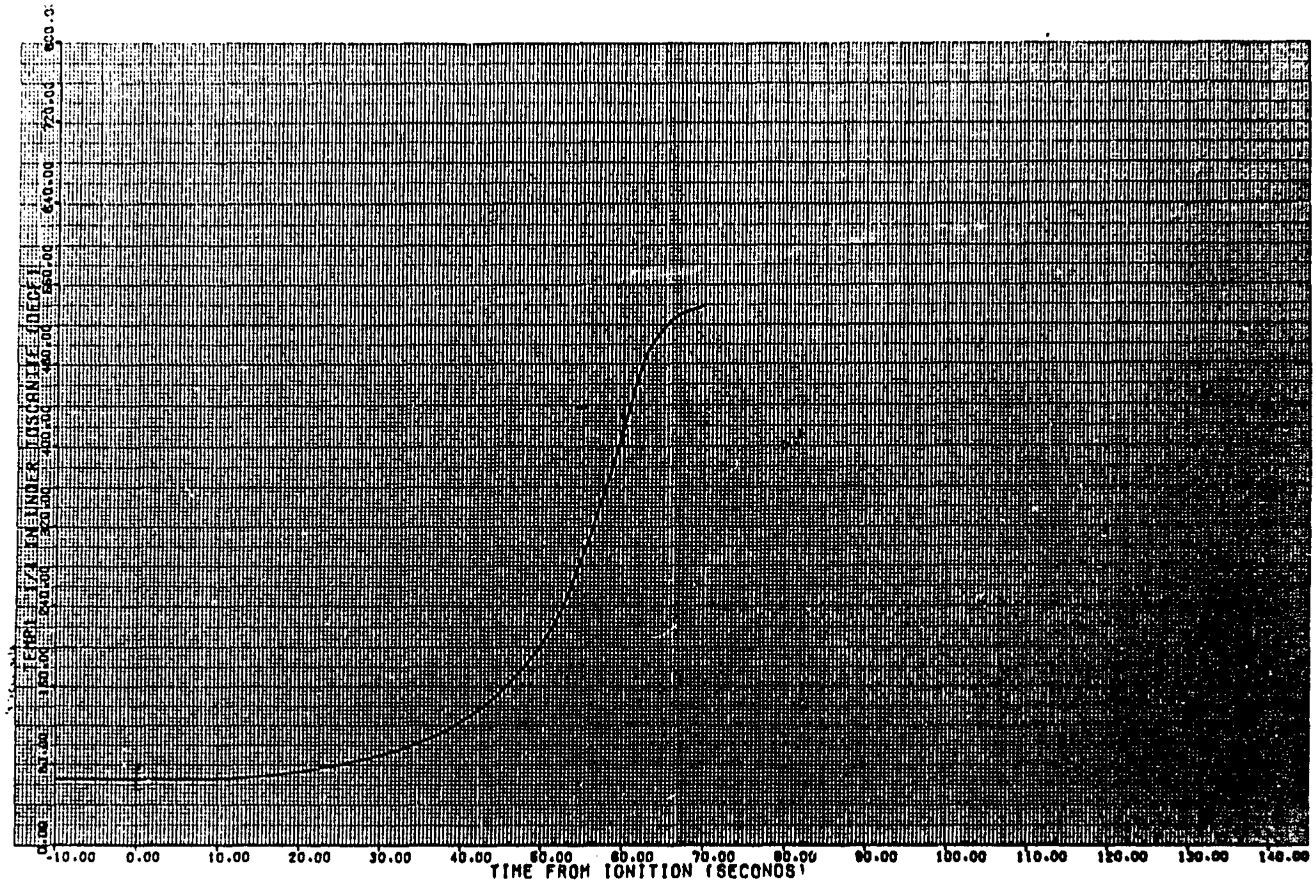

Figure 69. 1/4-inch Carbon Black Sample, Temperature-vs-Time Trace.

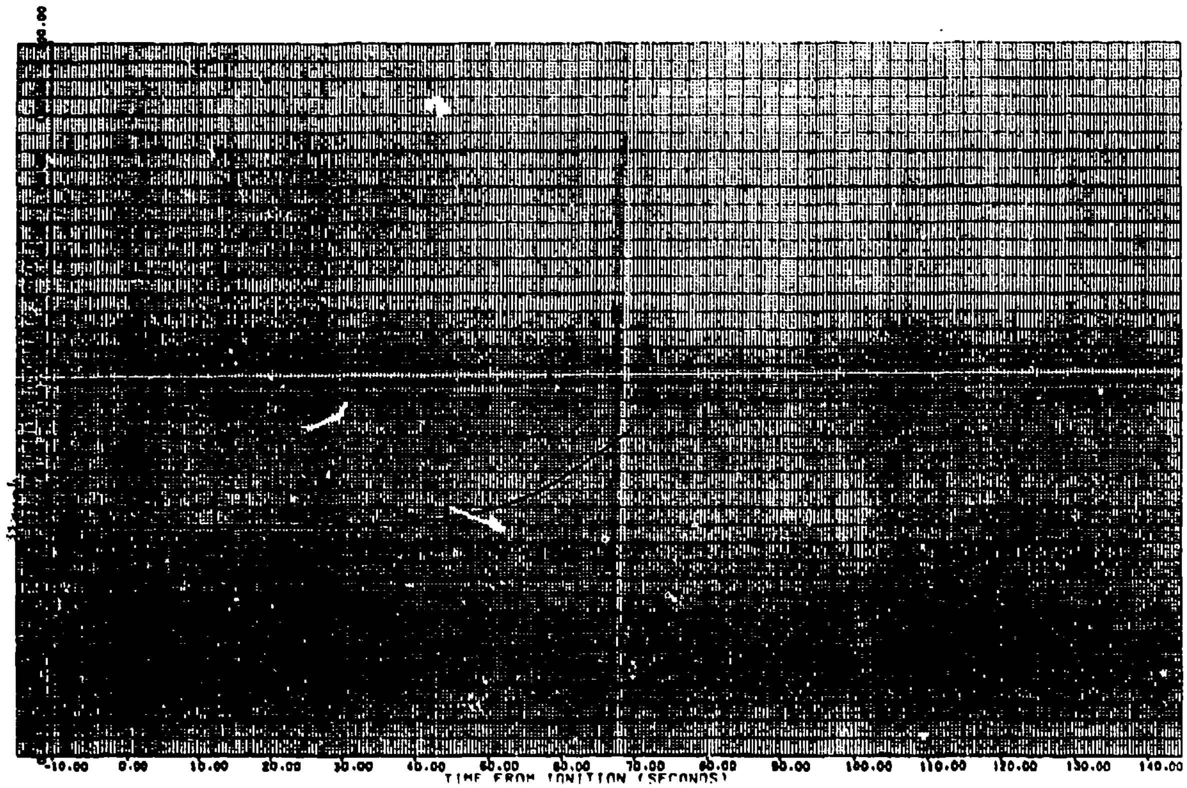

Figure 70. 1/2-inch Carbon Black Sample, Temperature-vs-Time Trace.

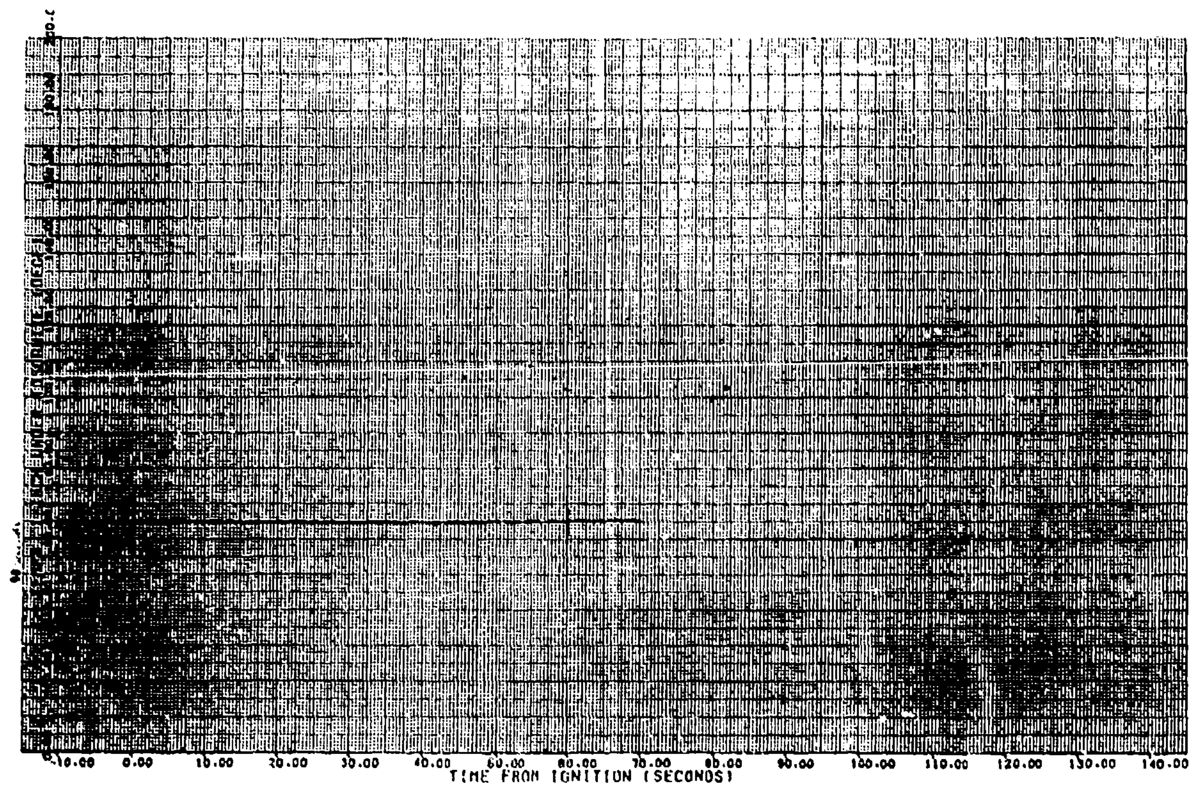

Figure 71. 1-inch Carbon Black Sample, Temperature-vs-Time Trace.

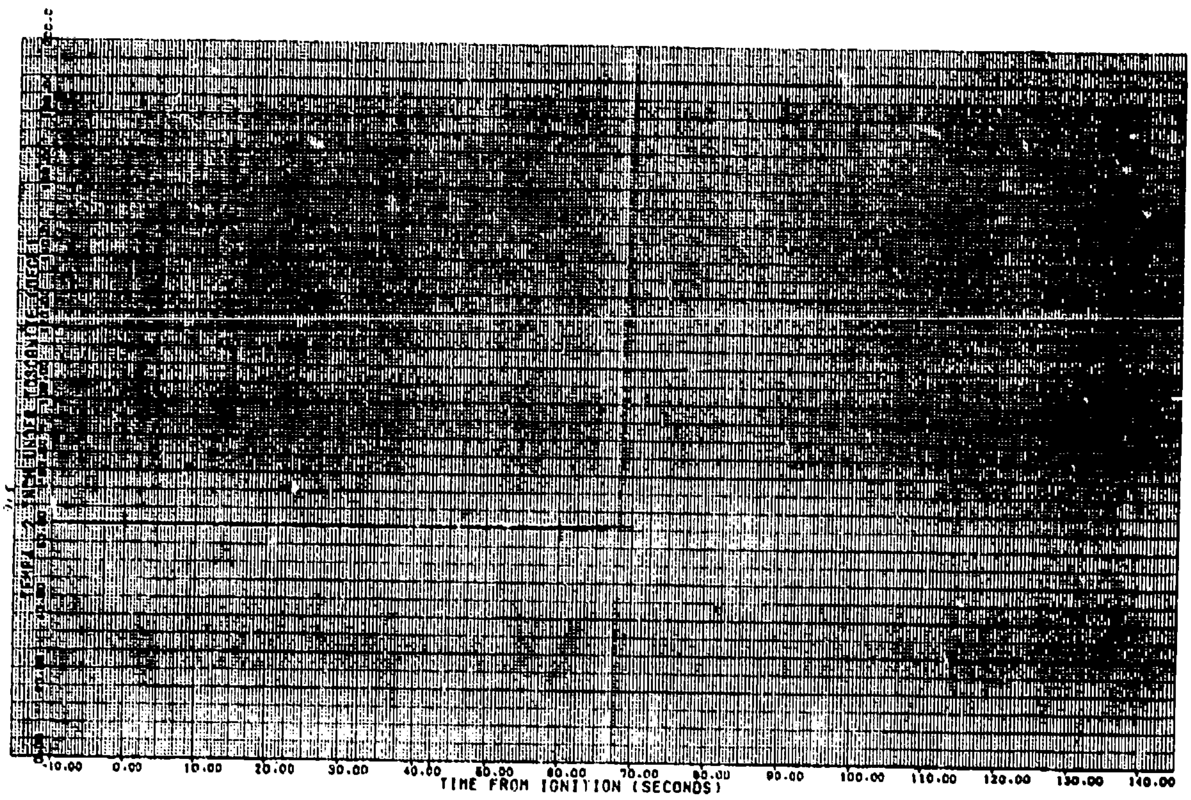

Figure 72. 2-inch Carbon Black Sample, Temperature-vs-Time Trace.

3.3.5. Altitude Diffuser Experiments.

The objective of this experiment was to compare the endurance of V-61, Dow-Corning's DC 93-058, and samples of sand and carbon black, which were all placed in direct line of the exhaust of a super BATES rocket motor. Samples of Dow-Corning's DC 93-058; V-61; samples that contained 42% carbon black, LIF, and CAO-14; and samples of 80%, 85%, and 90% sand were cast on the altitude diffuser's walls. The samples were placed side-by-side, approximately 24 inches from the exit cone of the super BATES motor. V-61 was placed around the samples at approximately the same thickness as the sample (see Figures 73, 74, and 75).

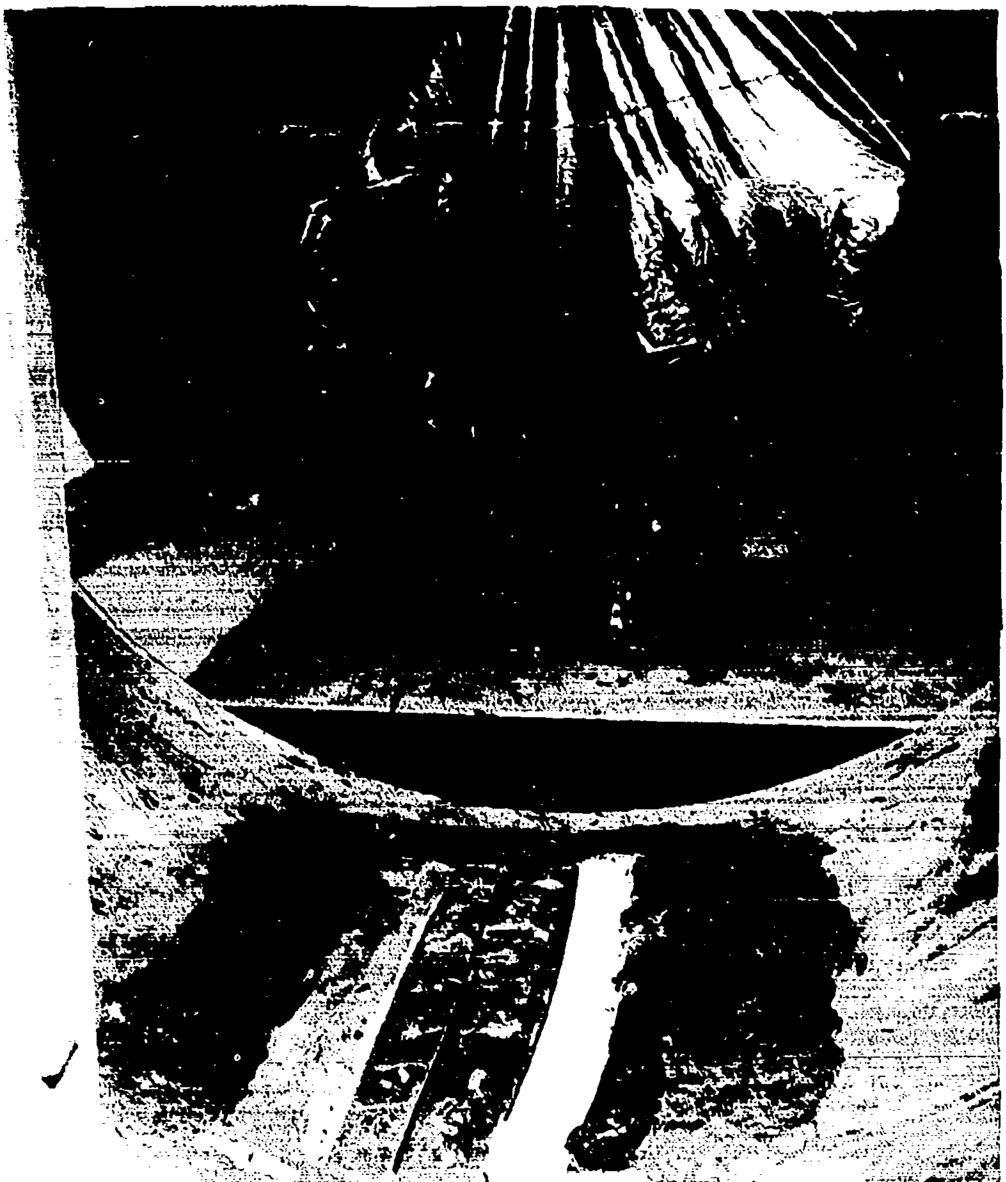

Figure 73. Sample Placement and Alignment, Altitude Diffuser.

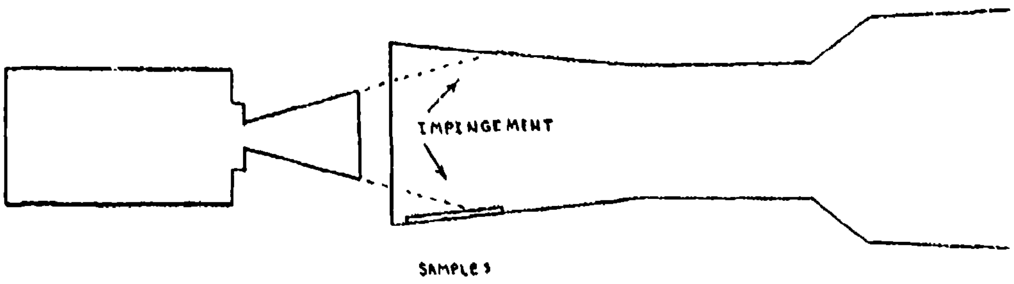

Figure 74. Motor and Diffuser Illustration.

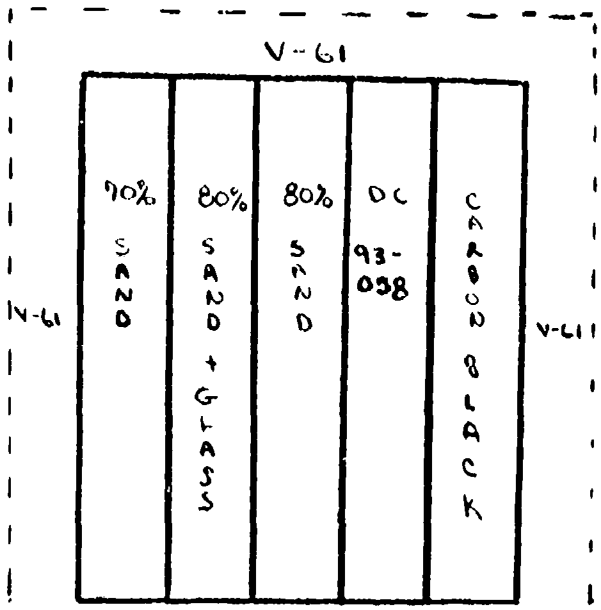

Figure 75. Sample on the Diffuser.

The firing lasted five seconds. After the firing, it was discovered that the Dow-Corning material debonded from the wall during the firing. Erosion, resulting from impingement on the sample, was evident. A depression 3 inches wide and 6 inches long was visible. It was felt that these results could be attributed to the fact that the material was beyond its shelf-life. The V-61 performed well; however, it is hazardous and expensive to use. The sand formulations performed well, but these formulations are too dense to apply to overhead surfaces. The carbon black sample showed very little char from the firing, and it is easy to apply (see Figure 76). The results showed the sand and carbon black samples were able to withstand the rocket motor exhaust as well as the V-61 and DC 93-058.

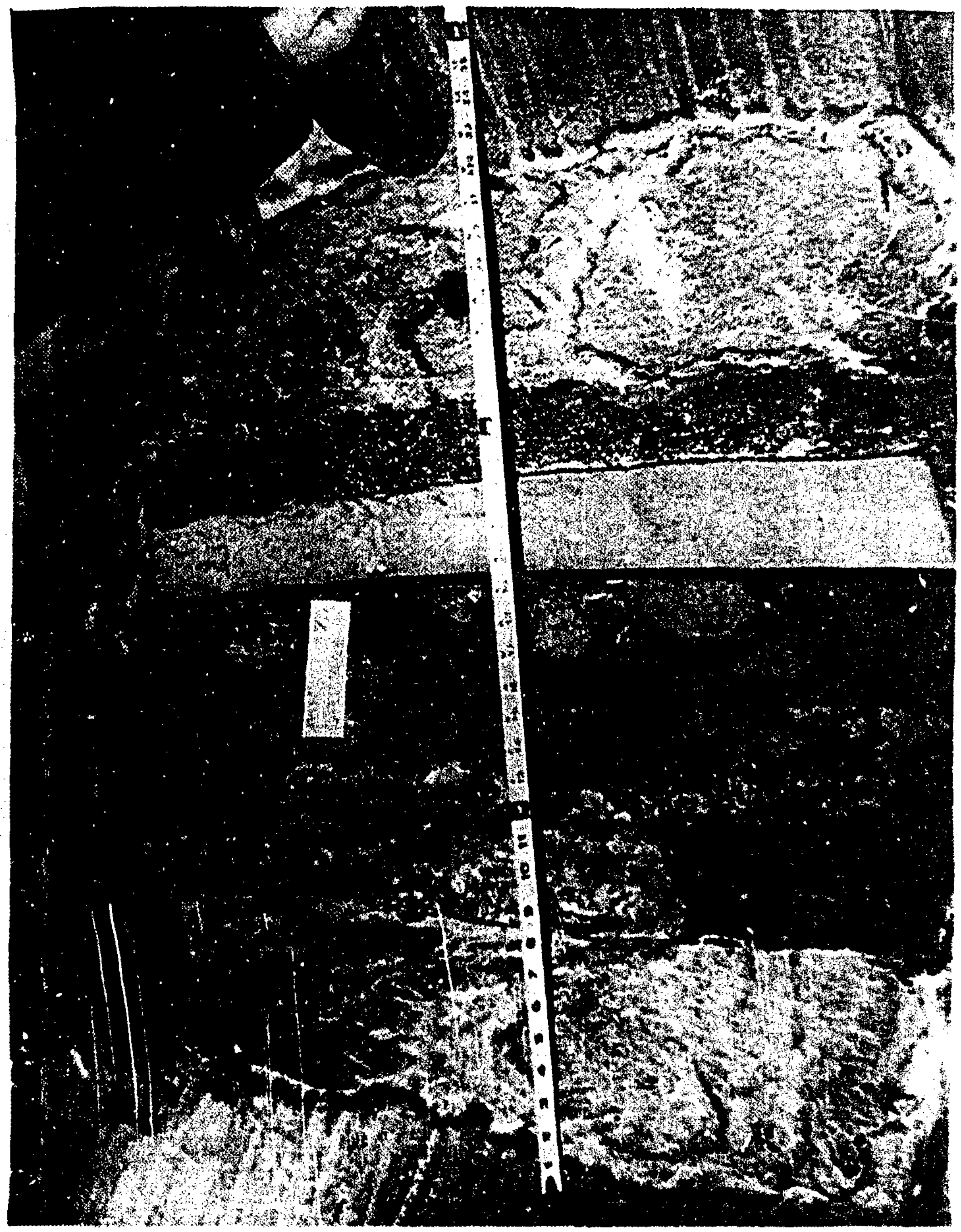

Figure 76. Post Results of Samples on the Diffuser.

3.3.6. Space Shuttle Experiments.

Six samples were tested, four of sand, and two of HLM-85 graphite, using the STS-5 Space Shuttle Columbia's exhaust. The samples were cast on carbon steel plates, 1/8-inch by 6-inches by 6-inches, cured at the AFRPL, and then transported to Florida. The formulations of the sand samples were 70%, 80%, 85%, and 90% sand. The graphite formulations contained 70% and 80% graphite (see Figures 77 through 82). The samples were mounted on carrier plates with other samples the Kennedy Space Center (KSC) was evaluating. The carrier plates were mounted on the mobile launch platform (see Figure 83).

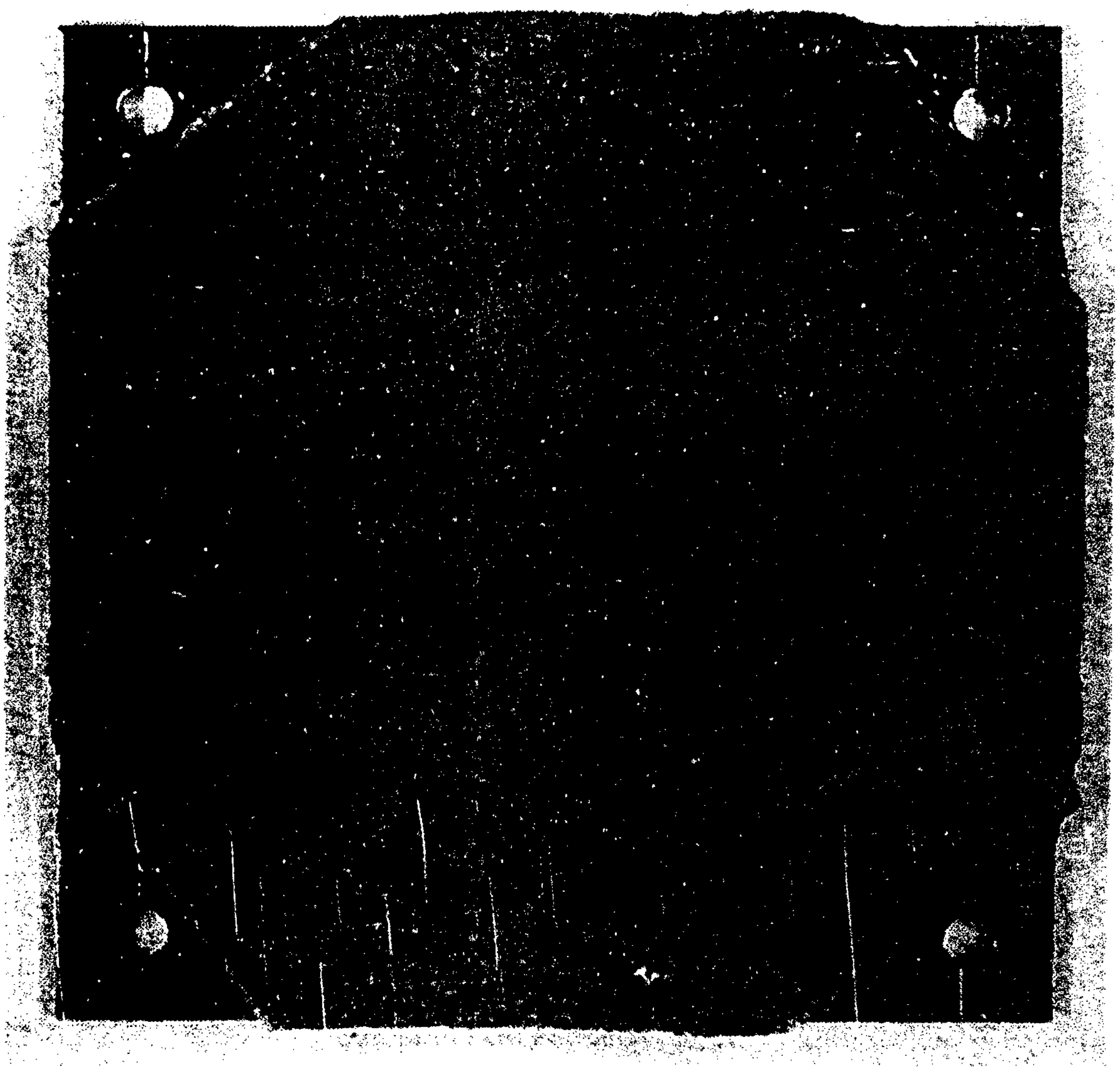

Figure 77. 80% Sand Sample, KSC Pre Photograph.

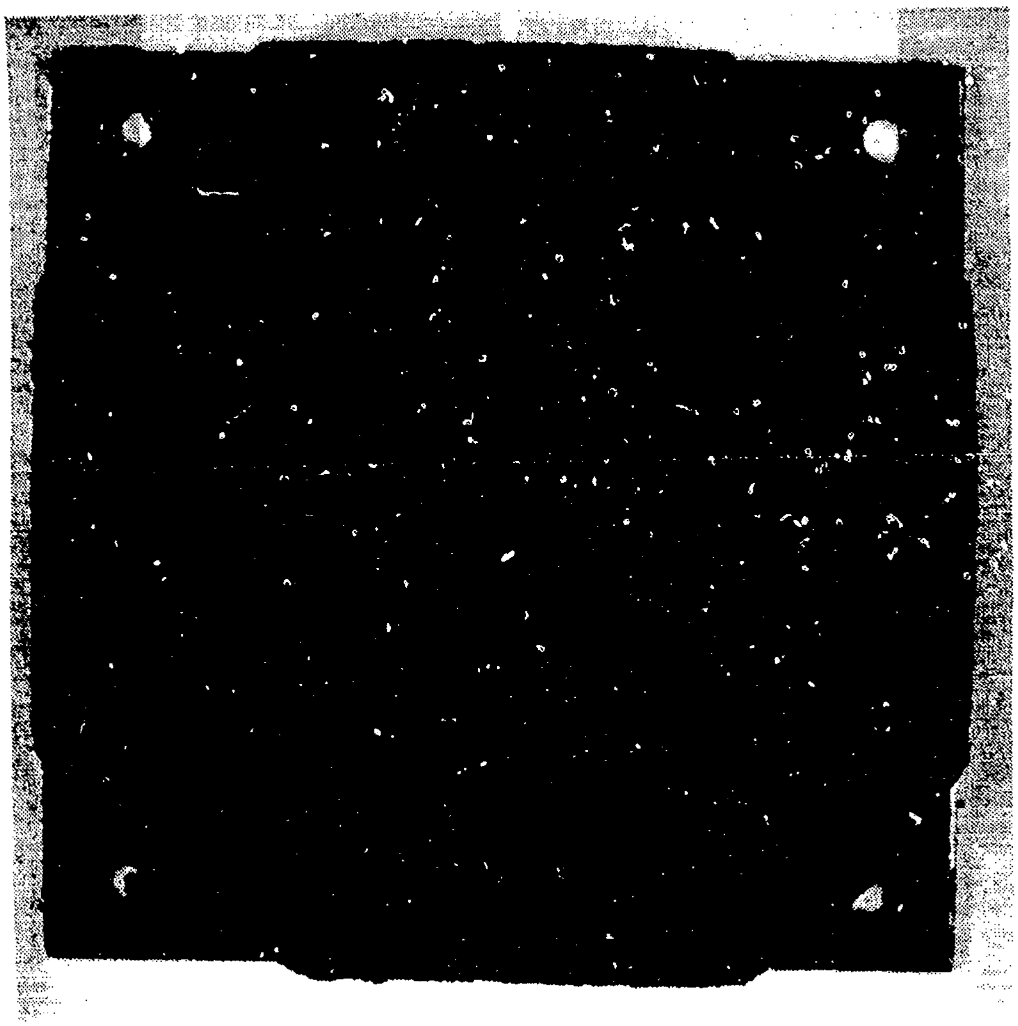

Figure 78. 80% Graphite Sample, KSC Pre Photograph.

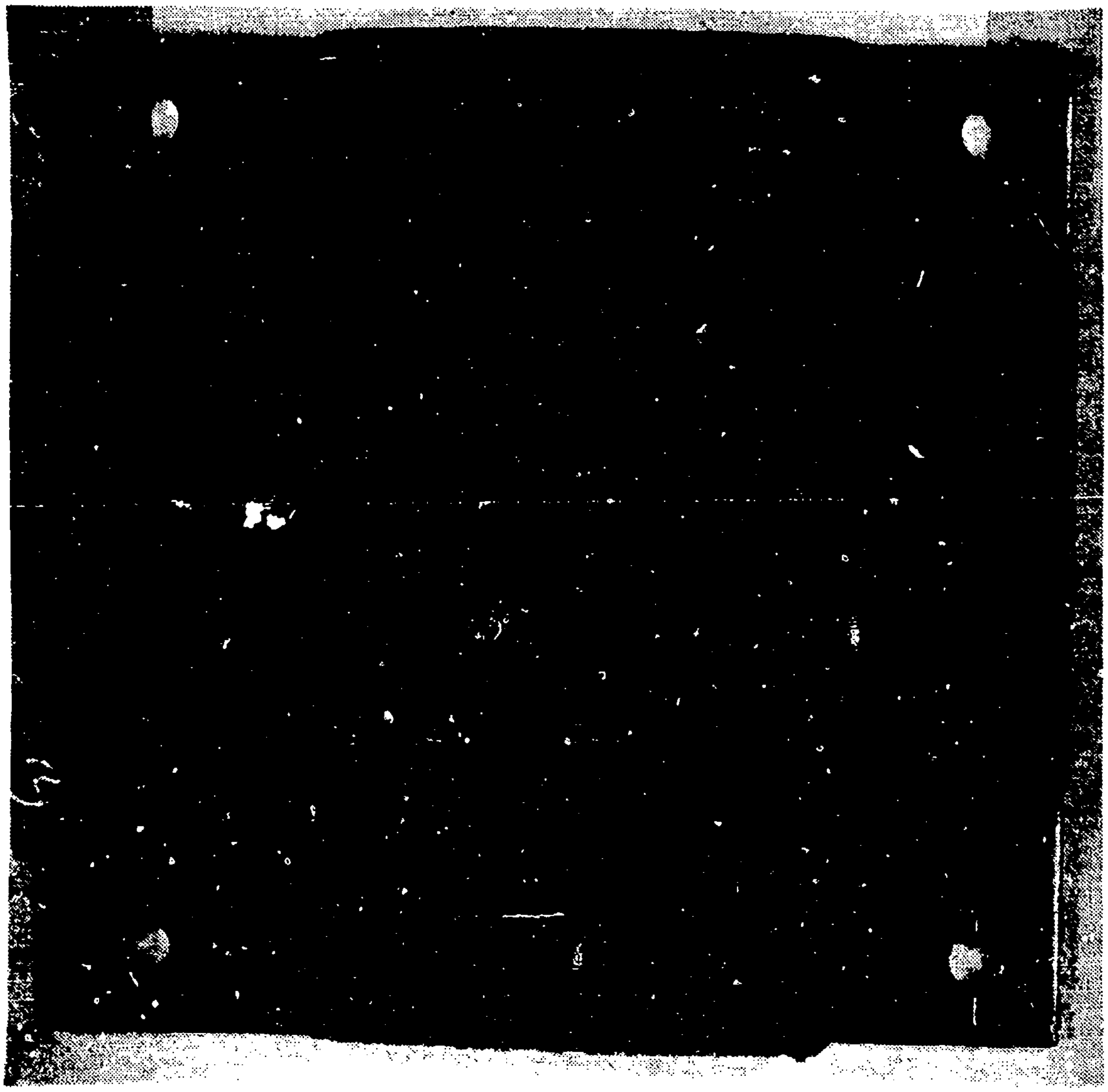

Figure 79. 85% Sand Sample, KSC Pre Photograph.

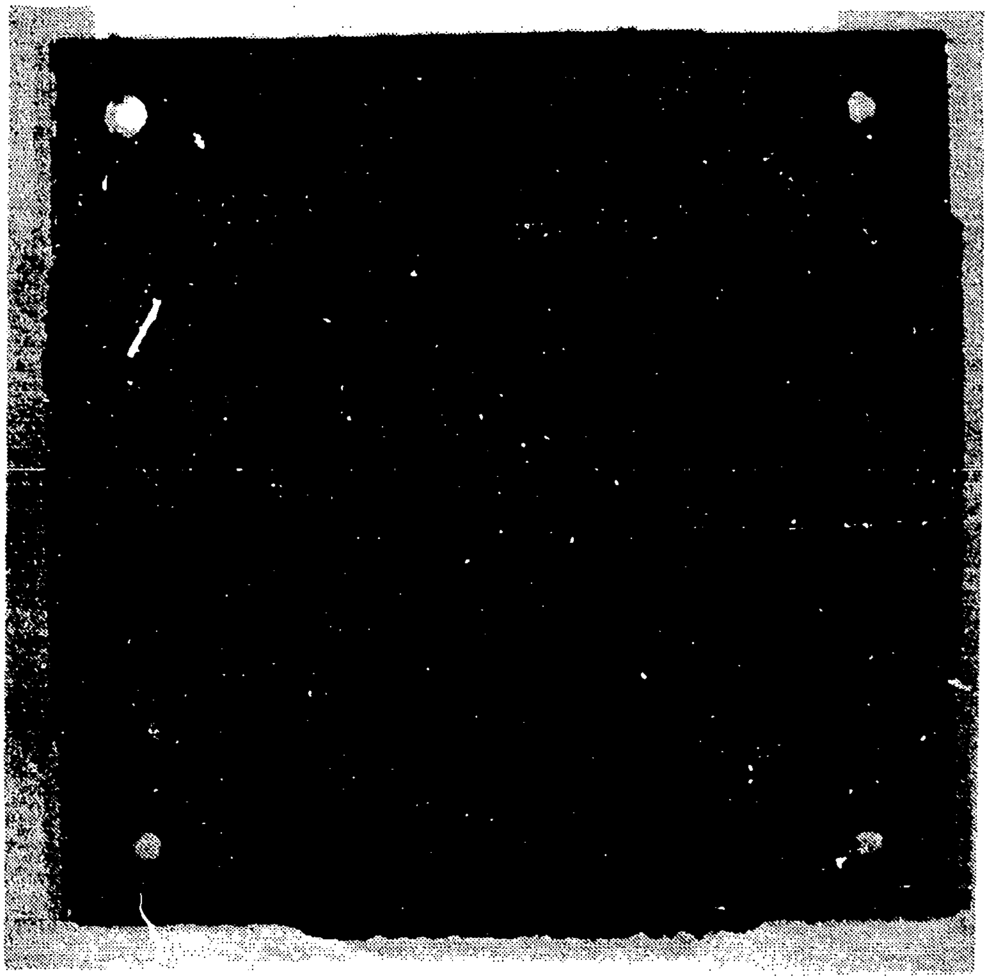

Figure 80. 70% Graphite Sample, KSC Pre Photograph.

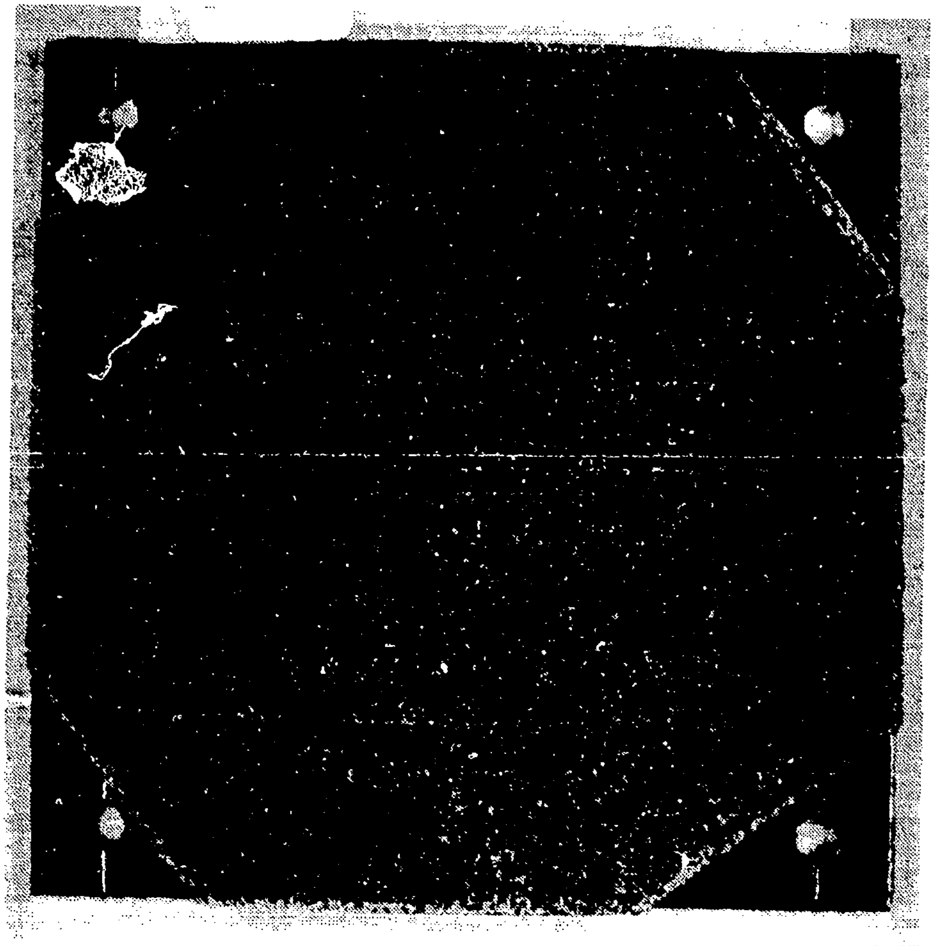

Figure 81. 90% Sand Sample, KSC Pre Photograph.

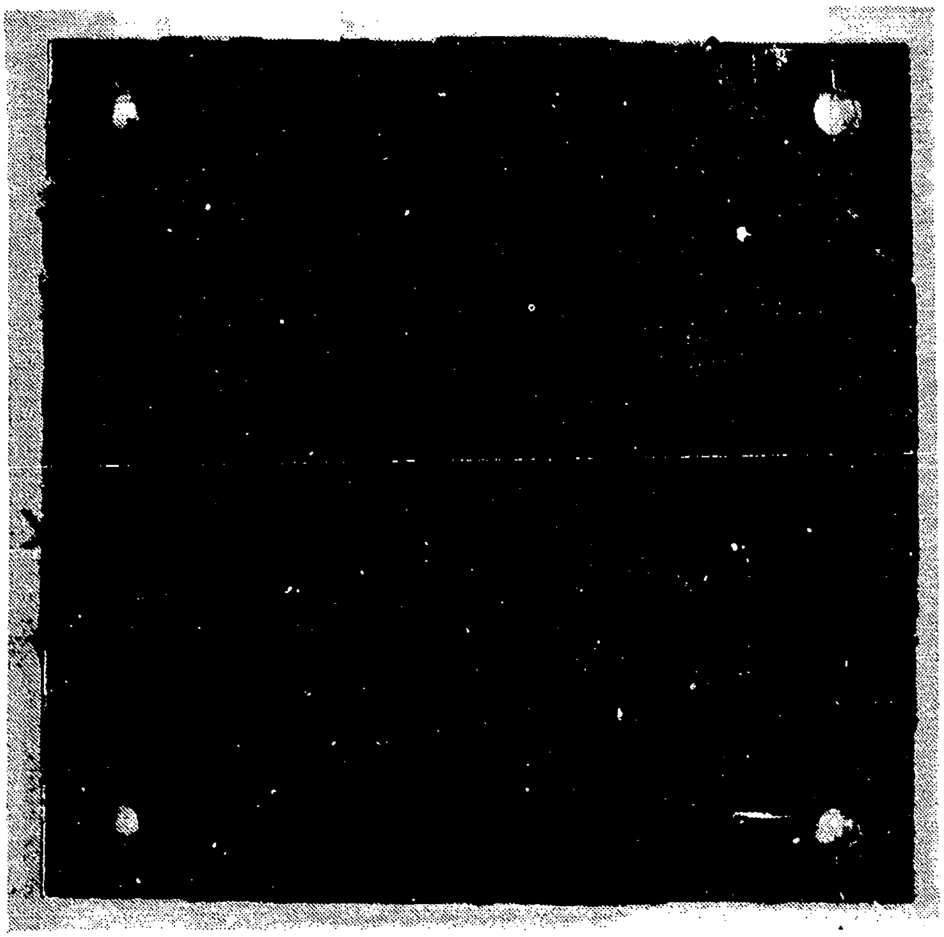

Figure 82. 70% Sand Sample, KSC Pre Photograph.

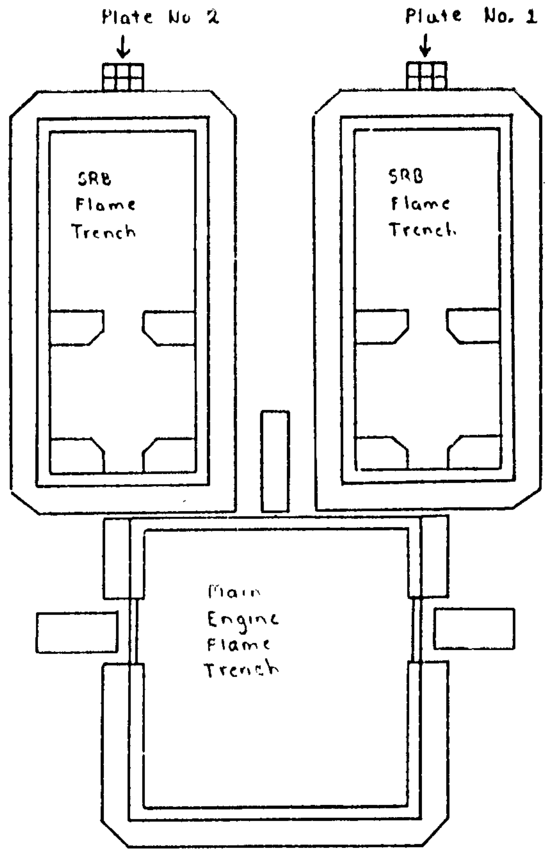

Figure 83. Mobile Launch Platform and Carrier Plate Placement.

After the launch, the AFRPL's samples were evaluated, using the same calculation for determining thickness and weight loss that was used by the KSC (see Table 6). The following is an example calculation:

<u>Panel # 15</u>

Thickness before exposure:	0.143 inch
Weight before exposure:	102 grams
Thickness after exposure:	0.074 inch
Weight after exposure:	49 grams

$$\% \text{ Thickness Loss} = \frac{0.143-0.074}{0.143} \times 100 = 48.3 \qquad \text{(EQ. 3.3.6-1)}$$

$$\% \text{ Weight Loss} = \frac{102-49}{102} \times 100 = 52.0 \qquad \text{(EQ. 3.3.7-2)}$$

Table 6. Results of Samples, Space Shuttle.

Material	Loss % Thickness	Loss % Weight	Temp (^{o}F)
80% Sand	61.6	65.6	150
80% Graphite	48.9	49.2	150
85% Sand	62.3	68.0	150
70% Graphite	53.0	55.5	150
90% Sand	62.2	69.3	150
70% Sand	55.3	56.1	150

As shown by the results of the evaluations, the AFRPL's samples survived the firing and performed their assigned task of protecting the steel plate (see Figures 84 through 89 for post-firing photographs of the six samples).

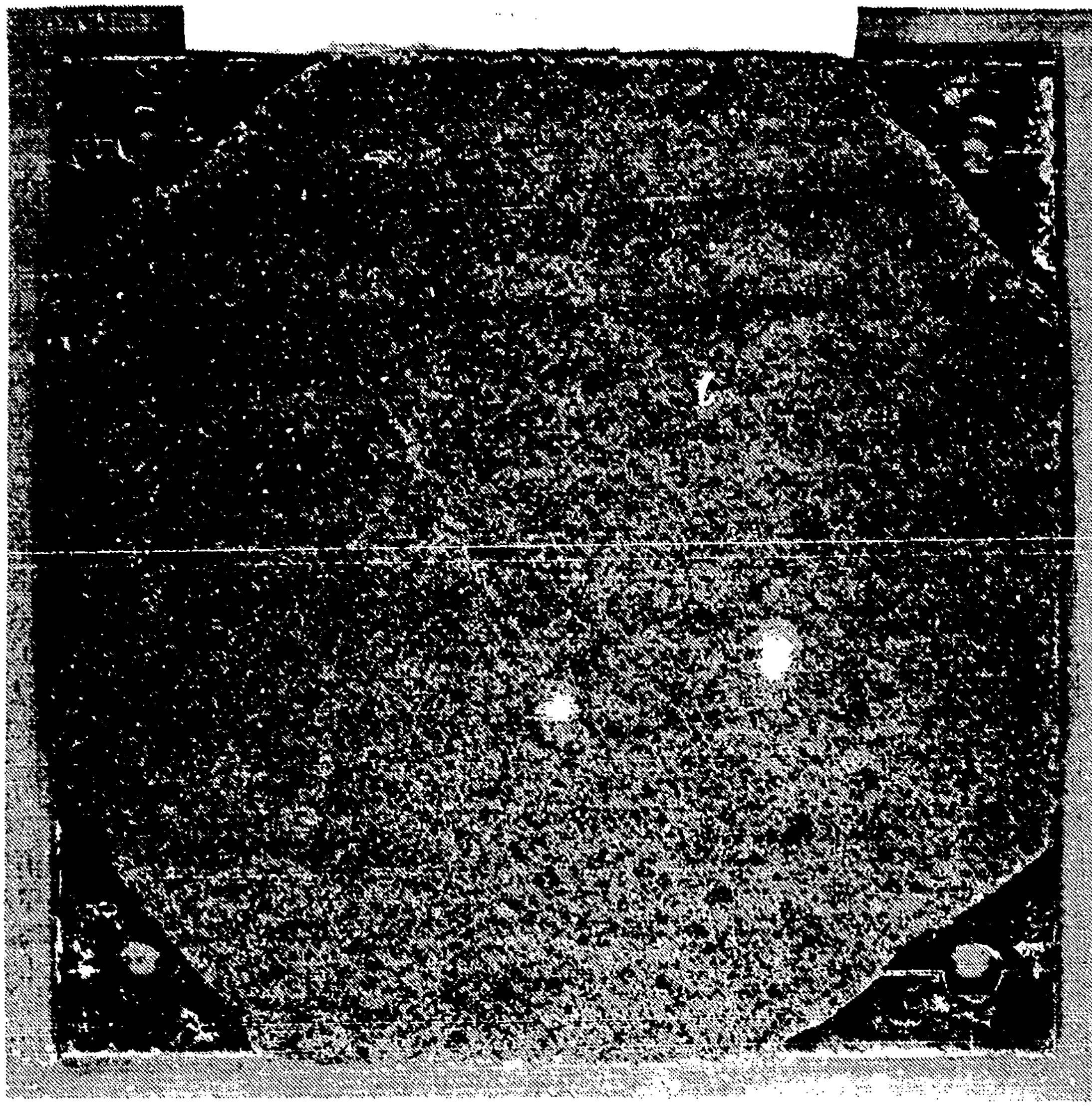

Figure 84. 80% Sand Sample, KSC Post Photograph.

Figure 85. 80% Graphite Sample, KSC Post Photograph.

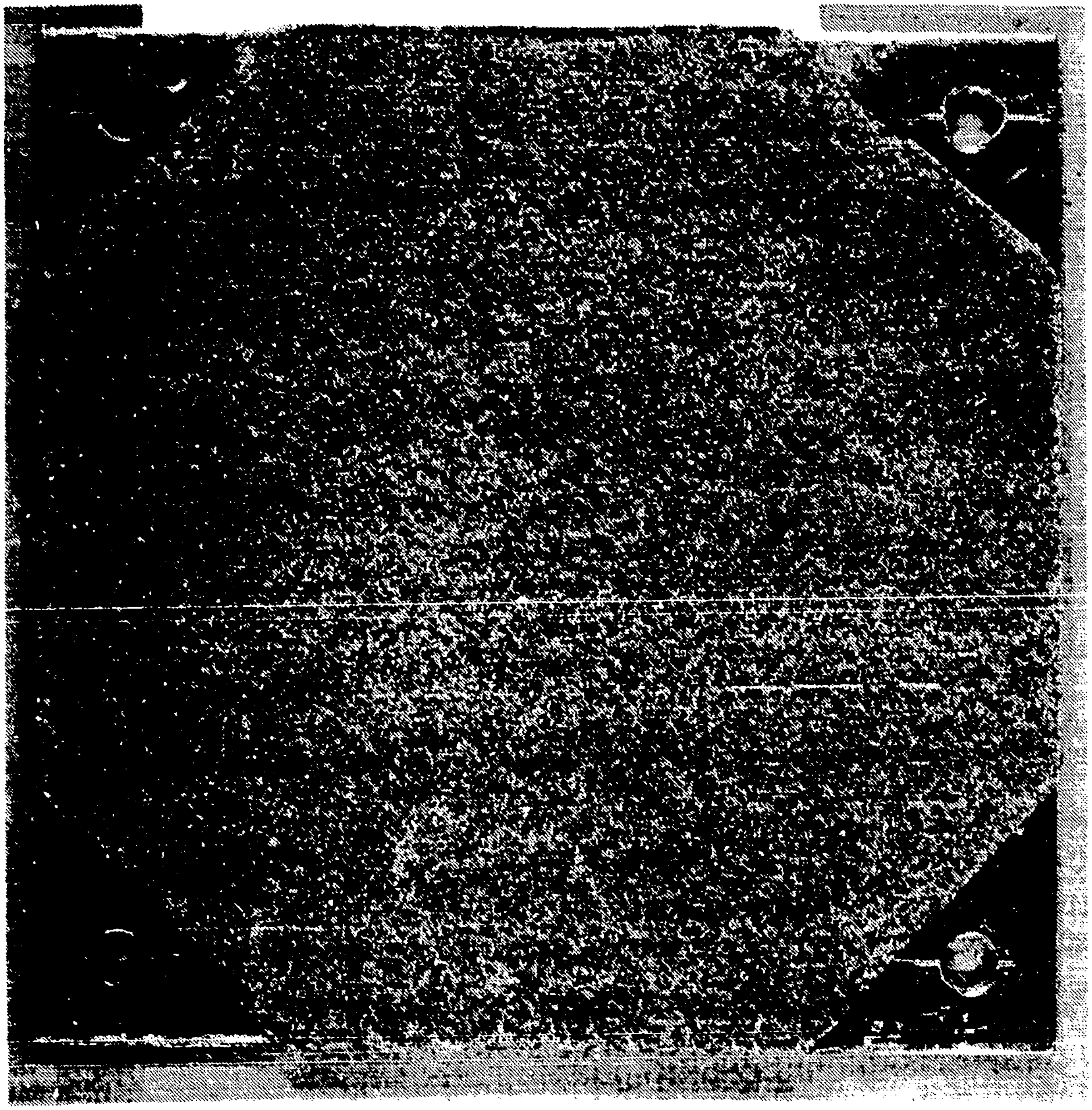

Figure 86. 85% Sand Sample, KSC Post Photograph.

Figure 87. 70% Graphite Sample, KSC Post Photograph.

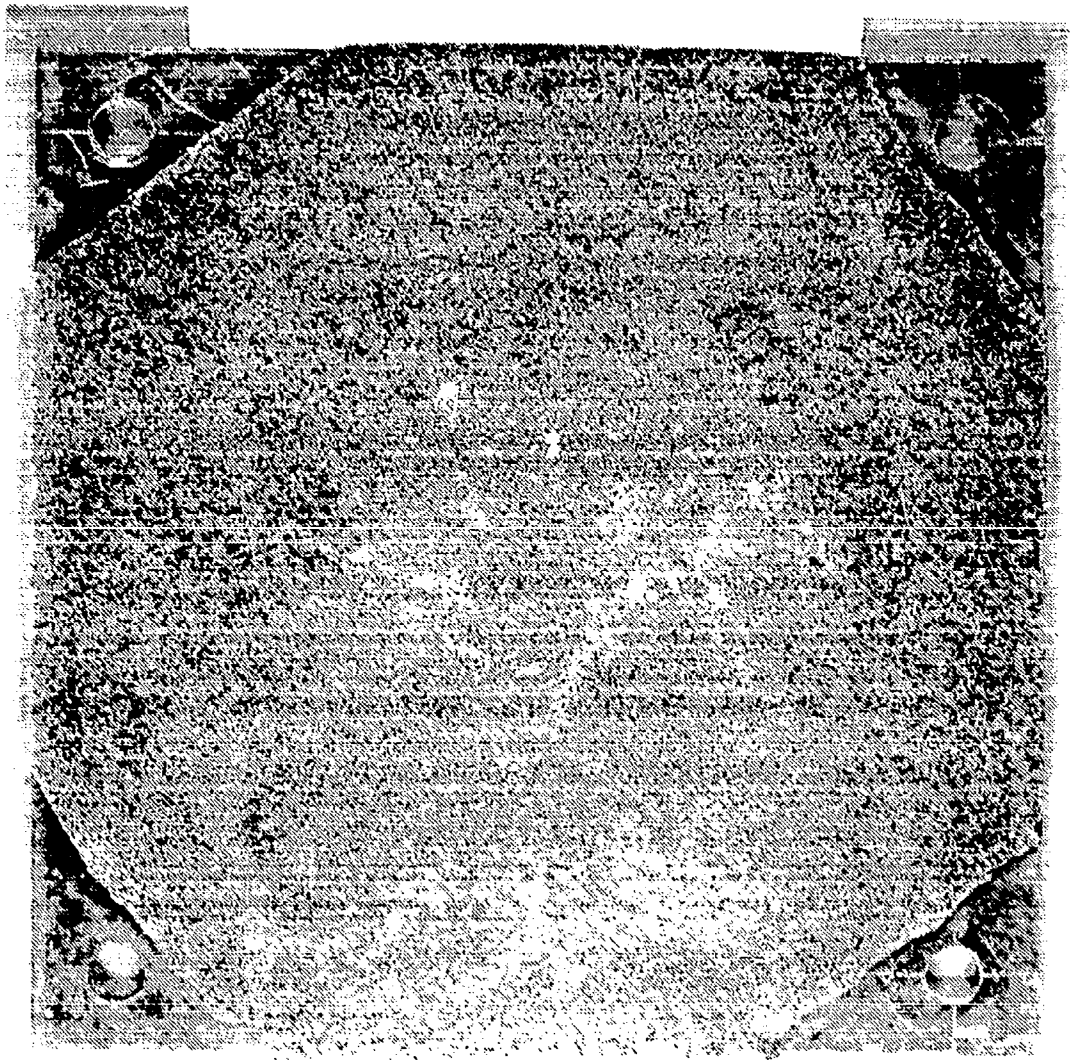

Figure 88. 90% Sand Sample, KSC Post Photograph.

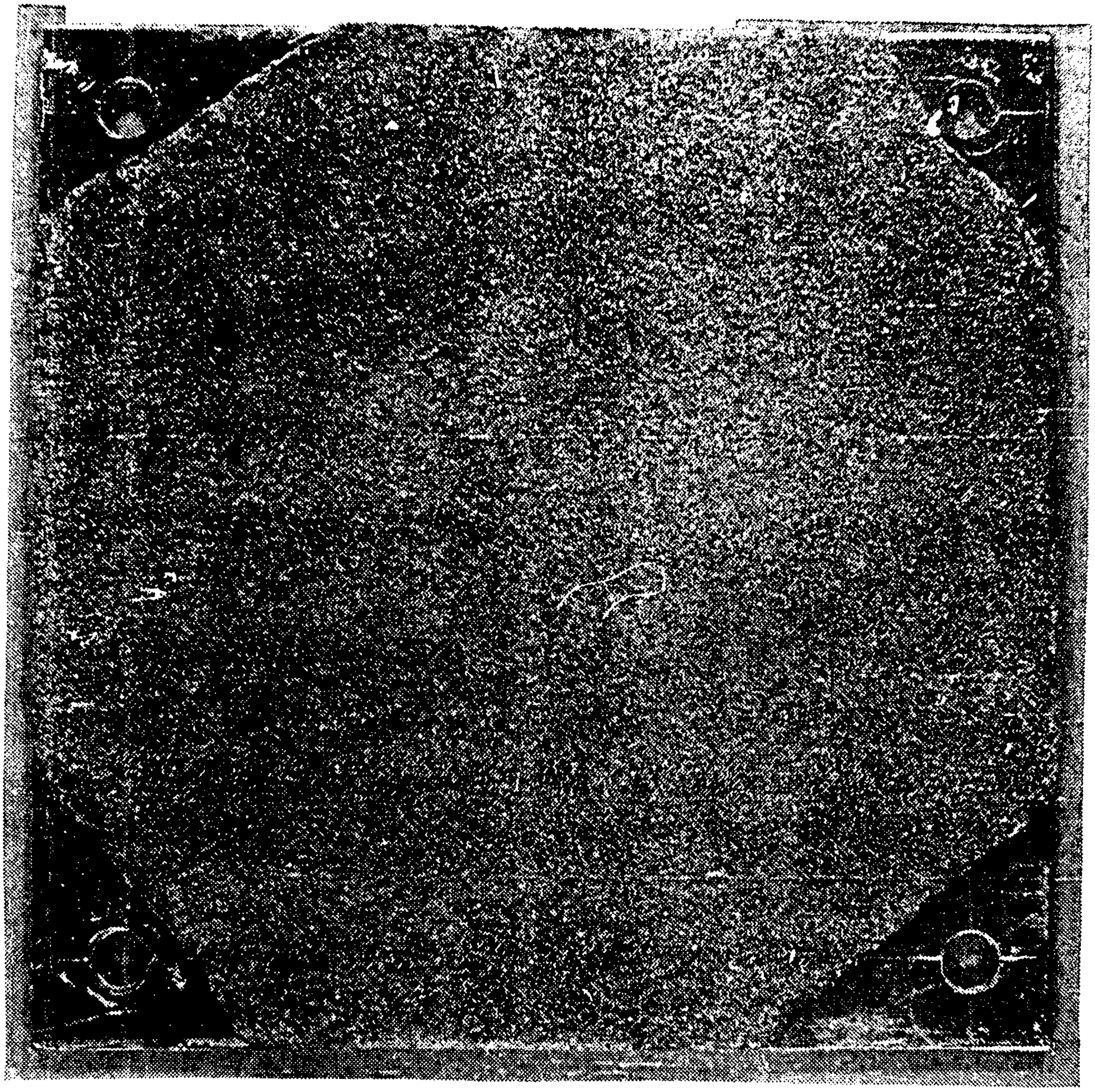

Figure 89. 70% Sand Sample, KSC Post Photograph.

4. CONCLUSIONS AND RECOMMENDATIONS.

4.1 Final Formulations.

The objectives of this project were to develop a formulation that would produce an effective, non-hazardous, low-cost insulation and one that would process easily in field conditions. This final goal was accomplished. Two successful products were selected from the many formulations, a medium-grit, silica sand mixture (Sand Toscanite) and carbon black (Carbon Black Toscanite).

Both materials proved to be a good insulator. Experiments showed the silica sand allowed very low rates of heat conduction, and the Carbon Black Toscanite gave more protection.

The author has applied for a patent in the name of the United States Air Force.

As each formulation is best suited for different applications, a description of the characteristics of each formulation follows.

Carbon Black Toscanite

Carbon Black Toscanite can be applied by trowel to any surface, as the viscosity can be varied by changing the amount of carbon black. A tough inelastic rubber is obtained when the viscosity is increased by higher solids loading. By decreasing the amount of solids, a relatively elastic rubber material is produced. Carbon Black Toscanite has excellent bonding properties, and it is recommended for overhead, as well as vertical, horizontal, or angular surfaces. It is compatible with many liquid fuels and oxidizers. Very little work is needed to refurbish a surface previously protected by Carbon Black Toscanite. Just wipe the remaining Toscanite surface clean with a cloth soaked in acetone, let the surface dry, and apply the next coat of Toscanite on top of the remaining Toscanite.

Sand Toscanite

The Sand Toscanite is also applied with a trowel. Because of its high density, this formulation is best suited for flat, horizontal surfaces. It is also excellent for patching concrete or other open areas that will be subjected to direct or radiant heat. The viscosity of this material can also be varied by changing the solids loading. The silica sand can be formulated to cure rock-hard, or it can be mixed to cure to a softer, more elastic state. This Toscanite is also compatible with various liquid fuels and oxidizers. Refurbishment procedures are identical to those of the carbon black formulation.

4.2 Cost Analysis of Toscanite.

Tests have shown that both Toscanite formulations provide insulating protection comparable to that provided by other commercial insulations. Toscanite, however, is less expensive than other materials presently available. The following detailed list itemizes and totals the costs of producing a one-gallon mixture of Toscanite containing 19%, 24%, 29%, 34%, and 89% filler (Appendix D). A list of sources for acquiring ingredients is also provided (see Appendix E).

4.3 Areas of Application.

Possible uses for Toscanite include the refurbishment of Minuteman silos, Peacekeeper launch facilities, and launch or static testing sites that require an effective, low-cost insulation. The AFRPL uses Toscanite wherever possible, as it is suitable for many practical insulating uses. Carbon Black Toscanite has been used to protect the aft dome of a Minuteman III Stage motor during a firing. This same formulation has replaced V-61 material in the super BATES firings here at the AFRPL. It has also been used to protect the burst disc of the super BATES.

GLOSSARY

AFRPL	Air Force Rocket Propulsion Laboratory, Edwards AFB, CA 93523
BATES	Ballistic Test and Evaluation System, a motor
CAO-14	methylene bis(tertiary butyl phenol), an antioxidant produced by Sherwin Williams
Char-Tek 59	AVCO's insulating product
DBTDL	dibutyltin dilaurate, a cure catylst
DC 93-058	Dow Corning's insulating product
FTOS	flight termination ordnance system
HIPPO	high-internal pressure-producing orifice
HLM-85	graphite
HTPB	hydroxy-terminated polybutadiene, a polymer binder
IPDI	isophorone diisocyanate, a cure agent
ips	inches-per-second
KSC	Kennedy Space Center, Florida
LiF	lithium fluoride, a flame suppressant
LO_2	liquid oxygen
MMH	methyl hydrazine
NCO/OH	isocyanate-to-hydroxyl ratio
N_2H_4	hydrazine
N_2O_4	nitrogen tetroxide
NVC	no visible change
R45HT	ARCO Chemical Company's brand of HTPB
RP-1	kerosene-based rocket fuel
SiO_2	sand
UDMH	unsymmetrical dimethyl hydrazine
V-61	Kirkhill Rubber Corporation's insulating product, ME Hill-Gard V-61
μl	microliter

APPENDIX A. 50-GRAM MIXES AND HARDNESS TESTS.

Appendix A. 50-Gram Mixes and Hardness Tests.

Mix Number	Solids	NCO/OH	Density (grams/cubic centimeters)	Weight (grams)	Avg. Shore A
90-1	SiO_2	0.8	2.16	7.14	79
80-1	SiO_2	0.8	1.83	7.62	40
70-1	SiO_2	0.8	1.64	8.12	43
60-1	SiO_2	0.8			no cure
50-1	SiO_2	0.8	1.32	6.78	.5/20
90-2	SiO_2	0.9	2.12	7.79	88
80-2	SiO_2	0.9			no cure
70-2	SiO_2	0.9	1.64	7.28	22/48
60-2	SiO_2	0.9	1.47	6.68	9/56
50-2	SiO_2	0.9	1.33	6.14	7/49
90-3	SiO_2	1.0	2.14	7.30	94
80-3	SiO_2	1.0	1.78	7.80	73
70-3	SiO_2	1.0	1.63	7.88	30/66
60-3	SiO_2	1.0	1.47	7.06	22/83
50-3	SiO_2	1.0	1.34	7.14	16/64
90-2-1	Glass Beads	0.9	1.89	6.33	84
80-2-1	Glass Beads	0.9	1.73	7.28	11
70-2-1	Glass Beads	0.9			no cure
60-2-1	Glass Beads	0.9	1.43	7.05	.3/18
50-2-1	Glass Beads	0.9	1.31	6.31	1/17
90-3-1	Glass Beads	1.0	2.13	6.65	82
80-3-1	Glass Beads	1.0	1.65	6.90	83
70-3-1	Glass Beads	1.0	1.55	6.63	22/33
60-3-1	Glass Beads	1.0	1.48	6.60	28/67
50-3-1	Glass Beads	1.0	1.33	6.99	16/54
90-1-1	Glass Beads	0.8	2.05	6.99	94
80-1-1	Glass Beads	0.8	1.68	7.22	46
70-1-1	Glass Beads	0.8	1.62	7.04	6/6
60-1-1	Glass Beads	0.8	1.45	6.83	0/13
50-1-1	Glass Beads	0.8	1.32	6.43	10/12

APPENDIX B. FORMULATION OF 50-GRAM MIXES.

Appendix B. Formulation of 50-Gram Mixes.

Mix Number	90-1		
NCO/OH Ratio	0.8		
Polymer	R45HT	9.38%	4.69 grams
Curing Agent	IPDI	0.622%	0.311 grams
Solids	SiO_2	90%	45 grams
Cure Catalyst	DBTDL 10 μl		

Mix Number	80-1		
NCO/OH Ratio	0.8		
Polymer	R45HT	18.75%	9.34 grams
Curing Agent	IPDI	1.24%	0.622 grams
Solids	SiO_2	80%	40 grams
Cure Catalyst	DBTDL 10 μl		

Mix Number	70-1		
NCO/OH Ratio	0.8		
Polymer	R45HT	28.13%	14.07 grams
Curing Agent	IPDI	1.86%	0.933 grams
Solids	SiO_2	70%	35 grams
Cure Catalyst	DBTDL 10 μl		

Mix Number	60-1		
NCO/OH Ratio	0.8		
Polymer	R45HT	37.51%	18.75 grams
Curing Agent	IPDI	2.49%	1.24 grams
Solids	SiO_2	60%	30 grams
Cure Catalyst	DBTDL 10 μl		

Mix Number	50-1		
NCO/OH Ratio	0.8		
Polymer	R45HT	46.89%	23.44 grams
Curing Agent	IPDI	3.11%	1.56 grams
Solids	SiO_2	50%	25 grams
Cure Catalyst	DBTDL 10 μl		

Mix Number	90-2		
NCO/OH Ratio	0.9		
Polymer	R45HT	9.31%	4.65 grams
Curing Agent	IPDI	0.694%	0.347 grams
Solids	SiO_2	90%	45 grams
Cure Catalyst	DBTDL 10 μl		

Mix Number	80-2		
NCO/OH Ratio	0.9		
Polymer	R45HT	18.61%	9.31 grams
Curing Agent	IPDI	1.39%	0.694 grams
Solids	SiO_2	80%	40 grams
Cure Catalyst	DBTDL 10 μl		

Mix Number	70-2		
NCO/OH Ratio	0.9		
Polymer	R45HT	27.91%	13.95 grams
Curing Agent	IPDI	2.10%	1.04 grams
Solids	SiO_2	70%	35 grams
Cure Catalyst	DBTDL 10 μl		

Mix Number	60-2		
NCO/OH Ratio	0.9		
Polymer	R45HT	37.22%	18.61 grams
Curing Agent	IPDI	2.78%	1.38 grams
Solids	SiO_2	60%	30 grams
Cure Catalyst	DBTDL 10 μl		

Mix Number	50-2		
NCO/OH Ratio	0.9		
Polymer	R45HT	46.52%	23.26 grams
Curing Agent	IPDI	3.47%	1.73 grams
Solids	SiO_2	50%	25 grams
Cure Catalyst	DBTDL 10μl		

Mix Number	90-3		
NCO/OH Ratio	1.0		
Polymer	R45HT	9.23%	4.61 grams
Curing Agent	IPDI	0.766%	0.383 grams
Solids	SiO_2	90%	45 grams
Cure Catalyst	DBTDL 10 µl		

Mix Number	80-3		
NCO/OH Ratio	1.0		
Polymer	R45HT	18.47%	9.233 grams
Curing Agent	IPDI	1.53%	0.766 grams
Solids	SiO_2	80%	40 grams
Cure Catalyst	DBTDL 10 µl		

Mix Number	70-3		
NCO/OH Ratio	1.0		
Polymer	R45HT	27.7%	13.85 grams
Curing Agent	IPDI	2.30%	1.15 grams
Solids	SiO_2	70%	35 grams
Cure Catalyst	DBTDL 10 µl		

Mix Number	60-3		
NCO/OH Ratio	1.0		
Polymer	R45HT	36.93%	18.50 grams
Curing Agent	IPDI	3.06%	1 [illegible]3 grams
Solids	SiO_2	60%	30 grams
Cure Catalyst	DBTDL 10 µl		

Mix Number	50-3		
NCO/OH Ratio	1.0		
Polymer	R45HT	46.17%	23.08 grams
Curing Agent	IPDI	3.83%	1.91 grams
Solids	SiO_2	50%	25 grams
Cure Catalyst	DBTDL 10 µl		

Mix Number	90-2-1		
NCO/OH Ratio	0.9		
Polymer	R45HT	9.31%	4.65 grams
Curing Agent	IPDI	0.694%	0.347 grams
Solids	Glass Beads	90%	45 grams
Cure Catalyst	DBTDL 10 μl		

Mix Number	80-2-1		
NCO/OH Ratio	0.9		
Polymer	R45HT	18.61%	9.31 grams
Curing Agent	IPDI	1.39%	0.694 grams
Solids	Glass Beads	80%	40 grams
Cure Catalyst	DBTDL 10 μl		

Mix Number	70-2-1		
NCO/OH Ratio	0.9		
Polymer	R45HT	27.91%	13.96 grams
Curing Agent	IPDI	2.18%	1.04 grams
Solids	Glass Beads	70%	35 grams
Cure Catalyst	DBTDL 10 μl		

Mix Number	60-2-1		
NCO/OH Ratio	0.9		
Polymer	R45HT	37.22%	18.61 grams
Curing Agent	IPDI	2.78%	1.39 grams
Solids	Glass Beads	60%	30 grams
Cure Catalyst	DBTDL 10 μl		

Mix Number	50-2-1		
NCO/OH Ratio	0.9		
Polymer	R45HT	46.52%	23.26 grams
Curing Agent	IPDI	3.47%	1.74 grams
Solids	Glass Beads	50%	25 grams
Cure Catalyst	DBTDL 10 μl		

Mix Number	90-3-1		
NCO/OH Ratio	1.0		
Polymer	R45HT	9.23%	4.62 grams
Curing Agent	IPDI	0.766%	0.380 grams
Solids	Glass Beads	90%	45 grams
Cure Catalyst	DBTDL 10μl		

Mix Number	80-3-1		
NCO/OH Ratio	1.0		
Polymer	R45HT	18.47%	9.23 grams
Curing Agent	IPDI	1.53%	0.760 grams
Solids	Glass Beads	80%	40 grams
Cure Catalyst	DBTDL 10μl		

Mix Number	70-3-1		
NCO/OH Ratio	1.0		
Polymer	R45HT	27.2%	13.85 grams
Curing Agent	IPDI	2.30%	1.15 grams
Solids	Glass Beads	70%	35 grams
Cure Catalyst	DBTDL 10μl		

Mix Number	60-3-1		
NCO/OH Ratio	1.0		
Polymer	R45HT	36.93%	18.46 grams
Curing Agent	IPDI	3.06%	1.53 grams
Solids	Glass Beads	60%	30 grams
Cure Catalyst	DBTDL 10 μl		

Mix Number	50-3-1		
NCO/OH Ratio	1.0		
Polymer	R45HT	46.16%	23.08 grams
Curing Agent	IPDI	3.83%	1.92 grams
Solids	Glass Beads	50%	25 grams
Cure Catalyst	DBTDL 10 μl		

Mix Number	90-1-1		
NCO/OH Ratio	0.8		
Polymer	R45HT	9.38%	4.69 grams
Curing Agent	IPDI	0.620%	0.311 grams
Solids	Glass Beads	90%	45 grams
Cure Catalyst	DBTDL 10 μl		

Mix Number	80-1-1		
NCO/OH Ratio	0.8		
Polymer	R45HT	18.75%	9.38 grams
Curing Agent	IPDI	1.24%	0.620 grams
Solids	Glass Beads	80%	40 grams
Cure Catalyst	DBTDL 10 μl		

Mix Number	70-1-1		
NCO/OH Ratio	0.8		
Polymer	R45HT	28.13%	14.1 grams
Curing Agent	IPDI	1.86%	0.90 grams
Solids	Glass Beads	70%	35 grams
Cure Catalyst	DBTDL 10 μl		

Mix Number	60-1-1		
NCO/OH Ratio	0.8		
Polymer	R45HT	46.88%	23.44 grams
Curing Agent	IPDI	3.11%	1.55 grams
Solids	Glass Beads	60%	30 grams
Cure Catalyst	DBTDL 10 μl		

Mix Number	50-1-1		
NCO/OH Ratio	0.8		
Polymer	R45HT	46.88%	23.44 grams
Curing Agent	IPDI	3.11%	1.55 grams
Solids	Glass Beads	50%	25 grams
Cure Catalyst	DBTDL 10 μl		

Mix Number	80-10		
NCO/OH Ratio	1.0		
Polymer	R45HT	18.47%	9.23 grams
Curing Agent	IPDI	1.53%	0.766 grams
Solids	HLM 85 Graphite	80%	40 grams
Cure Catalyst	DBTDL 10µl		

Mix Number	90-5		
NCO/OH Ratio	0.9		
Polymer	R45HT	9.31%	4.65 grams
Curing Agent	IPDI	.694%	0.347 grams
Solids	Dolomite	90%	45 grams
Cure Catalyst	DBTDL 10µl		

APPENDIX C. FORMULATION OF 50-GRAM MIXES WITH ADDITIVES.

Appendix C. Formulation of 50-Gram Mixes with Additives.

Mix Number	80-1-(4-0)		
NCO/OH Ratio	1.5		
Polymer	R45HT	17.8%	8.9 grams
Curing Agent	IPDI	2.2%	1.1 grams
Solids	SiO_2	79.0%	39.5 grams
Antioxidant	CAO-14	1.0%	0.5 grams
Cure Catalyst	DBTDL 10 μl		

Mix Number	80-1-1.4-0		
NCO/OH Ratio	1.4		
Polymer	R45HT	18.3%	9.1 grams
Curing agent	IPDI	1.7%	0.9 grams
Solids	SiO_2	79.0%	39.5 grams
Antioxidant	CAO-14	1.0%	0.5 grams
Cure Catalyst	DBTDL 10 μl		

Mix Number	80-1-1.3-0		
NCO/OH Ratio	1.3		
Polymer	R45HT	18.4%	9.2 grams
Curing Agent	IPDI	1.6%	0.8 grams
Solids	SiO_2	79%	39.5 grams
Antioxidant	CAO-14	1%	0.5 grams
Cure Catalyst	DBTDL 10 μl		

Mix Number	80-1-1.2-0		
NCO/OH Ratio	1.2		
Polymer	R45HT	18.5%	9.25 grams
Curing Agent	IPDI	1.5%	.75 grams
Solids	SiO_2	9%	39.5 grams
Antioxidant	CAO-14	1%	0.5 grams
Cure Catalyst	DBTDL 10 μl		

Mix Number	80-1-1.1-0		
NCO/OH Ratio	1.1		
Polymer	R45HT	18.6%	9.3 grams
Curing Agent	IPDI	1.4%	0.7 grams
Solids	SiO_2	79%	39.5 grams
Antioxidant	CAO-14	1.0%	0.5 grams
Cure Catalyst	DBTDL 10 μl		

Mix Number	80-1-1-1		
NCO/OH Ratio	1.0		
Polymer	R45HT	18.6%	9.3 grams
Curing Agent	IPDI	1.40%	0.7 grams
Solids	SiO_2	80.0%	40.0 grams
Cure Catalyst	DBTDL 10 μl		

Mix Number	80-1-2-1		
NCO/OH Ratio	0.9		
Polymer	R45HT	18.6%	9.3 grams
Curing Agent	IPDI	1.40%	0.7 grams
Solids	SiO_2	80.0%	40.0 grams
Cure Catalyst	DBTDL 10 μl		

Mix Number	80-1-3-1		
NCO/OH Ratio	0.8		
Polymer	R45HT	18.8%	9.4 grams
Curing Agent	IPDI	1.2%	0.6 grams
Solids	SiO_2	80.0%	40.0 grams
Cure Catalyst	DBTDL 10 μl		

Mix Number	90-2-1-2		
NCO/OH Ratio	1.0		
Polymer	R45HT	9.2%	4.6 grams
Curing Agent	IPDI	0.8%	0.4 grams
Solids	Glass Beads	89.0%	44.5 grams
Antioxidant	CAO-14	1.0%	0.5 grams
Cure Catalyst	DBTDL 10 μl		

Mix Number	CB-3		
NCO/OH Ratio	1.3		
Polymer	R45HT	60%	30 grams
Curing Agent	IPDI	5%	2.5 grams
Solids	Carbon Black	34%	17 grams
Antioxidant	CAO-14	1%	0.5 grams
Cure Catalyst	DBTDL50 10 μl		

Mix Number	30-4-1		
NCO/OH Ratio	1.0		
Polymer	R45HT	65.6%	32.8 grams
Curing Agent	IPDI	4.4%	2.2 grams
Solids	Carbon Black	29%	14.5 grams
Antoixidant	CAO-14	1%	0.5 grams
Cure Catalyst	DBTDL 10 μl		

Mix Number	CB-2		
NCO/OH Ratio	1.3		
Polymer	R45HT	82.8%	41.4 grams
Curing Agent	IPDI	7.2%	3.5 grams
Solids	Carbon Black	9%	4.5 grams
Antioxidant	CAO-14	1%	4.5 grams
Cure Catalyst	DBTDL 10 μl		

APPENDIX D. TOSCANITE COSTS.

Appendix D. Toscanite Costs.

20% solids (19% filler; 1% antioxidant):

R45HT:	2884.07 gms = (106.92 oz) ($0.4/oz)	=	$	4.28
IPDI:	(353.93 gms) ($0.0175/gm)	=		6.19
CAO-14:	(40 gms) ($0.008/gm)	=		0.32
CB:	(760 gm) = 26.81 oz = (1.67 lb) ($0.56/lb)	=		0.10
			$	11.83/gallon*

25% solids (24% filler; 1% antioxidant):

R45HT:	2665.22 gms = (100.20) ($0.04/oz)	=	$	4.01
IPDI:	(332.78 gms) ($0.0175/gm)	=		5.83
CAO-14:	(40 gms) ($0.008/gm)	=		0.32
CB:	960 gm = 33.86 oz = (2.12 lb) ($0.56/lb)	=		1.19
DBTDL:	(2.0 gm) ($0.052/gm)	=		0.10
			$	11.45/gallon*

30% solids (29% filler; 1% antioxidant):

R45HT:	2488.34 gms = (93.55 oz) ($0.04/oz)	=	$	3.74
IPDI:	(310.58 gm) ($0.0175/gm)	=		5.44
CAO-14:	(40.0 gm) ($0.008/gm)	=		0.32
CB:	(1160 gm) = 40.92 oz = (2.56 lb) ($0.56/lb)	=		1.43
DBTDL:	(2.00 gm) ($0.052/gm)	=		0.10
			$	11.03/gallon*

35% solids (34% filler; 1% antioxidant):

R45HT:	2310.47 gm = (86.86 oz) ($0.04/oz)	=	$	3.47
IPDI:	(287.53 gm) ($0.0175/gm)	=		5.00
CAO-14:	(40 gm) ($0.008/gm)	=		0.32
CB:	1360 gm = 47.97 oz = (3 lb) ($0.56/lb)	=		1.68
DBTDL:	(2 gm) ($0.052/gm)	=		0.10
			$	10.57/gallon*

90% solids (89% filler; 1% antioxidant):

R45HT:	353.94 gm = (13.31 oz) ($0.04/oz)	=	$	0.53
IPDI:	(44.06 gm) ($0.0175/gm)	=		0.77
CAO-14:	(40 gm) ($0.008/gm)	=		0.32
Sand:	3560 gm = 3.56 kg = (7.85 lb) ($0.19/lb)	=		1.49
DBTDL:	(2 gm) ($0.052/gm)	=		0.10
			$	3.21/gallon*

*1 gallon = 4,000 grams.

APPENDIX E. SOURCES OF TOSCANITE INGREDIENTS.

Appendix E. Sources of Toscanite Ingredients.

R45HT	ARCO Chemical Company 1500 Market Street Philadelphia, Pennsylvania 19101 (215) 557-2286 $0.58/lb bulk $0.64/lb lots 50 drums
IPDI	Thorson Chemical Corporation 39 West 55th Street New York, New York 10019 (212) 421-0800 1 kg @$7.50
Carbon Black (Thermax)	R. T. Vanderbilt Company, Inc. 30 Winfield Street Norward, Connecticut 06855 (203) 853-1400 West Coast: 6279 East Slawson Avenue Los Angeles, California 90040 (213) 723-5208 2,000 pounds and less: $0.56 per pound 2,000 pounds and more: $0.51 per pound 50-pound bag container: $0.56 = $28.00 per bag $0.51 = $25.50 per bag
CAO-14	Sherwin Williams Chemicals P. O. Box 6520 Cleveland, Ohio 44101 (614) 889-3333 $3.60/lb